THE JOURNAL
OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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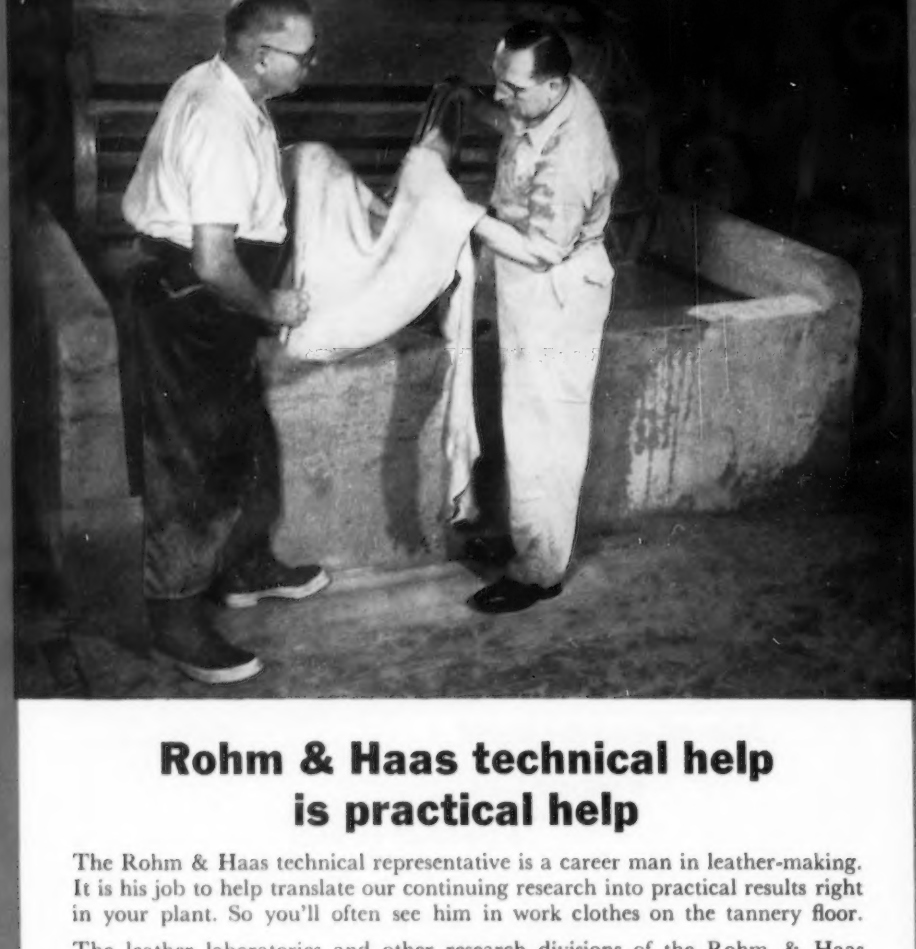

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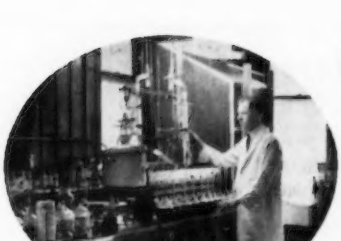

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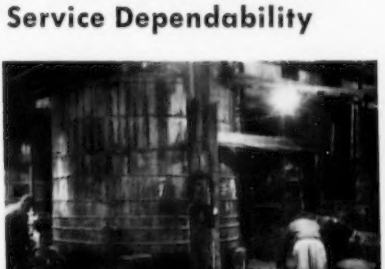

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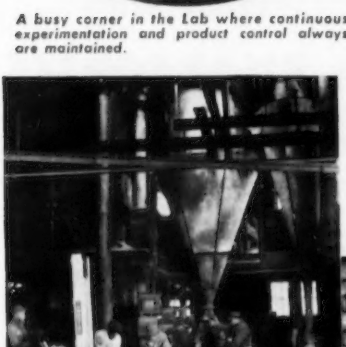

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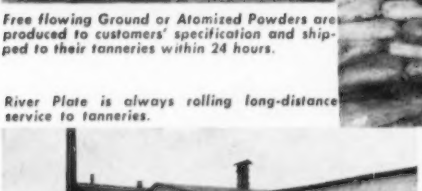

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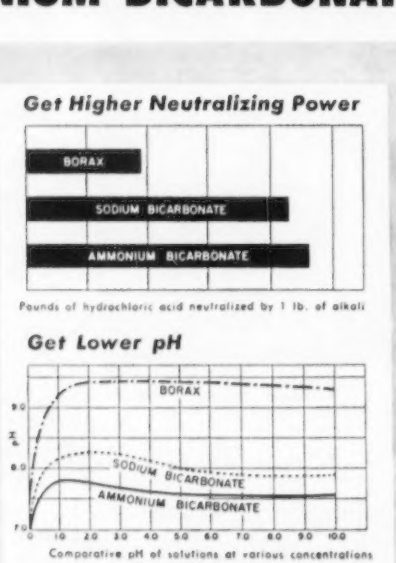

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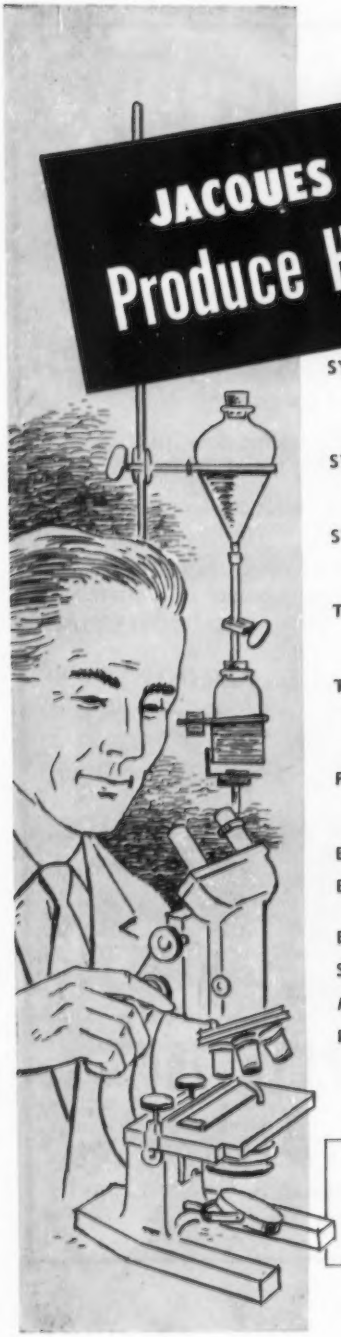

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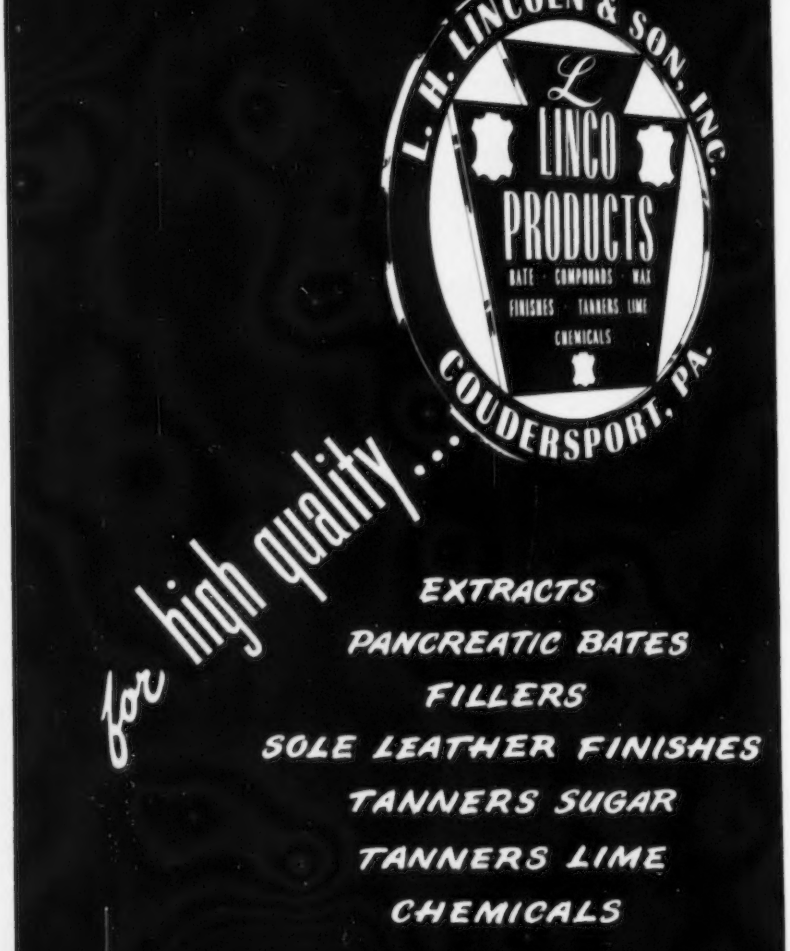
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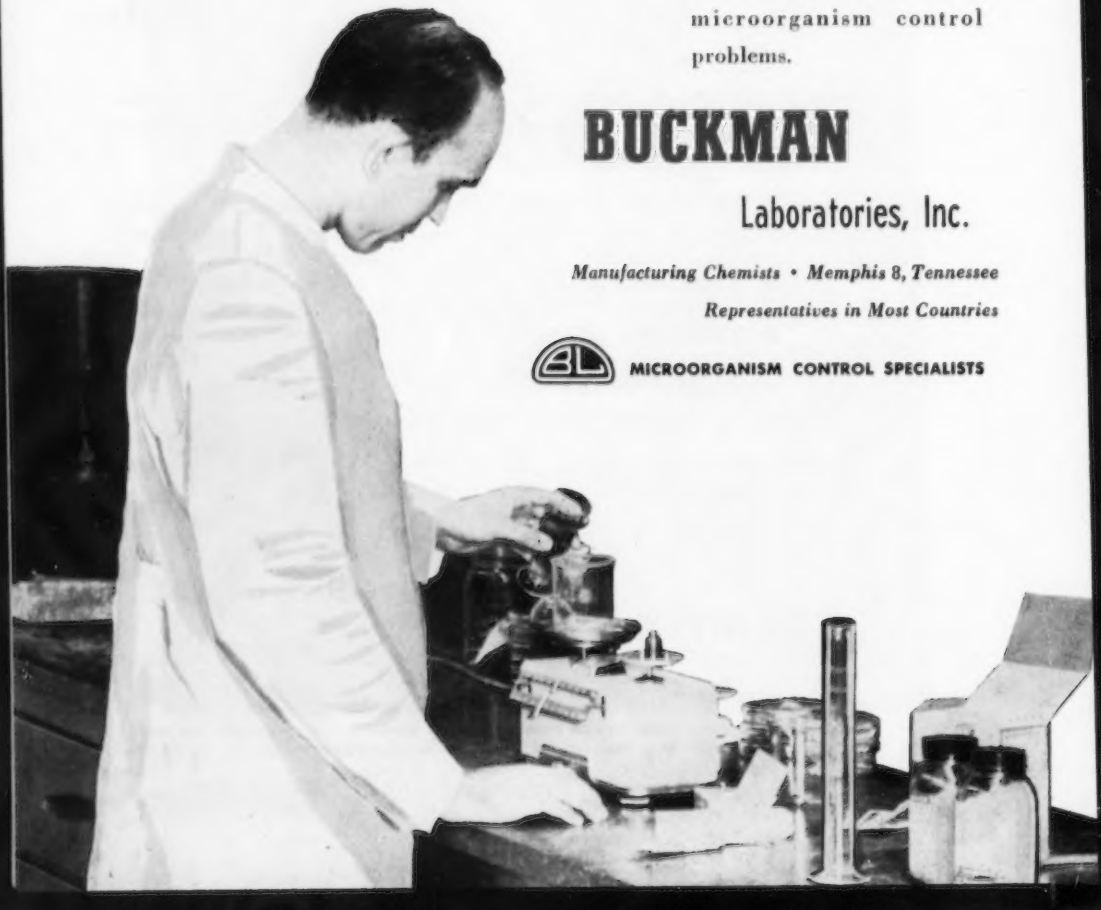

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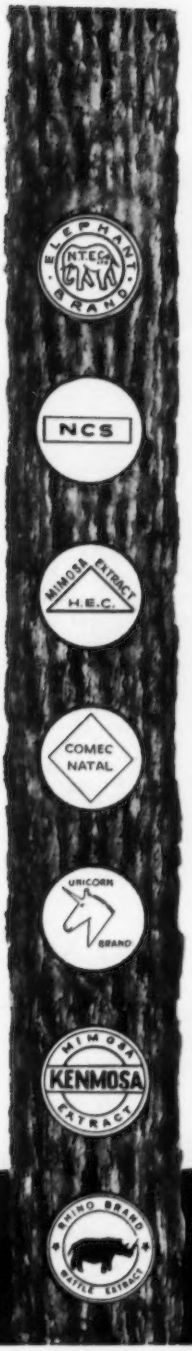

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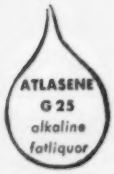

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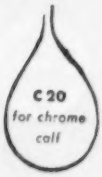

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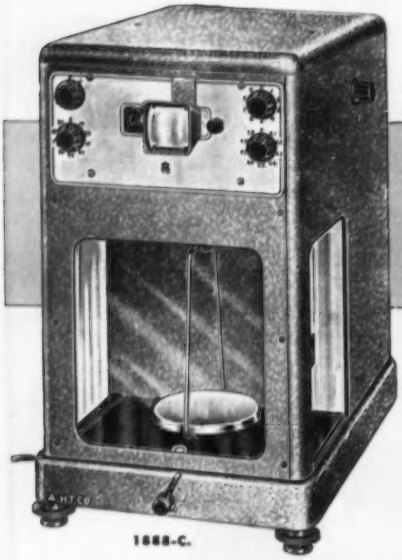

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JOURNAL OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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Article V - 2, Nominations (c)
Additional candidates may be nominated by petitions, signed by 15 or more active members. Such petitions shall reach the Secretary not later than April 1st, and shall contain a statement of the willingness of the nominee to serve if elected. No member shall sign more than one petition for each office to be filled.

INTERNATIONAL UNION OF LEATHER CHEMISTS' SOCIETIES FOURTH BIENNIAL CONFERENCE

STOCKHOLM, SWEDEN

August 1-4, 1955

H. G. TURLEY,
Research Associate
Rohm & Haas Company
Philadelphia, Pa.

As the "Viscount" flew over the beautiful city of Stockholm glimmering in the setting sun of a fine summer day, this introduction proved to be a happy augury of a wonderful meeting of leather scientists on the occasion

of the Fourth Biennial Conference of the International Union of Leather Chemists' Societies. For the conference, our hosts were the Swedish section of the Nordic Leather Chemists Society assisted by the Swedish Tanners Federation, the Swedish Chemical Society and the Allied Industries.

The conference began with a reception Sunday evening given by the Swedish section of the Nordic Leather Chemists' Society and we were welcomed by Mr. Kurt Hjort of the Swedish organizing committee. The reception was held in the Tekniska Museet (Museum of Technology). This fine science museum contains many of the past contributions of Sweden to the world in science and engineering. The director of the museum welcomed us and graciously made available to the visitors some private sections of the museum. We recall that one of the unique exhibits we saw were old automobiles of the early days of the century complete with their solid rubber tires, shiny brass horns and fine leather covered seats and cushions. This leather and finish was still in excellent condition, apparently as good as on the day it was first used forty or fifty years ago, but what a far cry from todays' beautiful automobiles upholstered in leather in all the colors of the rainbow and ranging from black to white.

The conference meetings were held in the fine lecture rooms of the Royal Institute of Technology where the Nobel Prize lectures are given. Accustomed as we are to the use of microphones and much electronic equipment, the acoustics here were a revelation, by speaking in an ordinary voice, the speaker could be heard plainly out to the fringes of the audience.

The conference opened Monday morning, August 1 with an address of welcome by the President, Dr. K. H. Gustavson, Director of the Swedish Tanning Research Institute. He welcomed the distinguished visitors; Dr. Sven Brohult, President of the Swedish Chemical Society, Mr. F. Moller, President of the Swedish Tanners Federation and the distinguished chemistry professor from the ancient University of Uppsala. The President also expressed the pleasure of the organizing committee at the large delegation of the overseas visitors, paying a kindly tribute to the group from the American Leather Chemists' Association who had made the long journey to participate in this meeting.

We then paid our silent respects to the members who had passed away since the last meeting, namely Professor Meunier, Professor McCandish, Dr. Cheshire and Mr. Paniker.

The conference was then addressed by Professor Stiasny who reminisced over the early days and the accomplishments of leather chemistry and officially opened the conference. Dr. Stiasny was well and still vigorous and sent, through me, his regards to his friends in the United States.

Dr. Gustavson then delivered the Presidential paper, taking for his topic "A Swedish Pioneer of Leather Technology in the Berzelian Era". He

referred to Carl Hylten-Cavallius (1817-1853) also known as Cavallin who successfully made leather with a mineral tannage of chrome and iron and patented this process in 1850, that is thirty-four years before the American Shultz introduced the two bath chrome tannage. Gustavson's paper contained many interesting points and should be read by every leather chemist.

Following the Presidential address, the Max Bergmann Memorial Lecture was given by Professor W. Grassmann, Director of the Max Planck Institute of Protein and Leather Research, under the title "Our Present Knowledge of Collagen". This lecture was of especial interest to those of us who knew Bergmann when he worked at the Rockerfeller Institute in New York. The wonderful protein collagen which is the basic material of our whole leather industry is becoming increasingly important in medical studies related to cancer, longevity and the life process of our bodies. After the Memorial Lecture, Grassmann was presented with a handsome Swedish glass vase bearing sculptured figures of leather workers. Grassman accepted this on behalf of his research institute.

Of two Symposia organized for the conference by the President, the first one on "The Physical Properties of Leather" was given in the afternoon under the Chairman, Mr. J. R. Blockey. Blockey pointed out that in the early days, the conference was concerned overwhelmingly with discussions of analytical methods for vegetable tans. It was of significance that at this conference the subject of leather would have the greatest attention. The four papers presented were of a high order including "Physical Properties of Leather" by Dr. R. J. Mitton of the British Leather Manufacturers Research Association. Mitton stresses the fact that leather is a product with a fibre structure which is modified by the tanner to produce the kind of leather required. The air spaces in leather have a great effect in promoting thermal insulation and particularly the transmission of water vapor which is so important for comfort in shoes and clothing leather.

Dr. E. Baumann, Director of Research of Bally Shoe Factories, Ltd., Schoenwerd, Switzerland gave a critical paper on "The Properties of Leather from the Shoe Manufacturing Point of View". Baumann was quite candid in admitting the greater use of synthetic materials to replace leather and warned us that leather must be studied more intimately and improved in a number of important properties. He hopes the International Union will plan an active part in promoting improvements in leather by the work of suitable committees. The physical testing of leathers must be advanced and improved and he gave a summary of the three groups of properties that are necessary for leather in shoes. He posed the question "What can we leather chemists do for leather?" and "What is needed is a bridge from science to practical tanning." In discussing waterproofing, Baumann mentioned three American processes, silicones, Dow - Quilon, duPont - Bavon of the Battelle Development Corpora-

tion. We need better colored leather. Some shades on the market are so poor that the leather would be better for printing photos than for making shoes. Leather must be perspiration proof because nylon socks don't absorb perspiration and there should be no sticking of the feet in unlined shoes. Dr. Baumann's plea was considered so urgent and timely that later in the conference a new commission of the Union was formed with Dr. R. G. Mitton as chairman to study and standardize physical methods for the examination of leather. We expect our most experienced members of the American Leather Chemists' Association to serve on this Committee. The sincerity of purpose behind this work is shown by Baumann's statement that "to reach agreement on certain methods of testing, it is a condition that personal and national prestige should be set aside in favor of the general interest in the further development of leather".

Then Dr. J. Kanagy of the National Bureau of Standards, Washington, presented his paper "The Properties of Leather; their Measurement and Establishing of Standards". Kanagy reviewed the properties of leather which make it useful for footwear and other wear. The development of methods of measuring these properties was discussed and it was explained how the results were used to establish specifications and standards in the United States and the relation of government and industry to these standards.

Finally, Professor Chambard, Director of the Research Insitute of the French Tanning School described the status of control of qualities in shoe leather for the consumer's use in France. Two physical test methods have found favor in France; the abrasion resistance determined by the application of an air stream of sand, and the softening of leather by treatment with water.

On Tuesday, an all day Symposium on "The Fundamentals of the Chemistry of Tanning Processes" was heard under the chairmanship of the President. Papers were delivered on the behavior of tans with proteins, chrome tanning, vegetable tanning, modern sole leather, synthetic tans including resin tannages, synthetic tans, dyes and auxiliaries, and the relationship of tannery processes.

Dr. K. G. A. Pankhurst of the B. L. M. R. A. described his work on studying the effect of tanning agents on the properties of thin surface films of collagen. This important work adds to our knowledge of the tanning mechanism by employing an entirely different and novel technique. Dr. D. Balanyi presented, at considerable length, "The Chrome Tanning Process", a review paper covering the literature of the chemistry of chrome tanning. Dr. T. White under "The Vegetable Tannage" presented a lecture giving a review of many of the hypotheses or ideas of the chemistry of vegetable tanning. He was followed by Mr. Holmes who described under "Some Aspects of the Modern Sole Leather Tannage", some of the recent advances that have been made in the tanning of sole leather.

Dr. A. Kuntzel, Director of the Institute of Tanning Chemistry at the Technical High School, Darmstadt reviewed the progress of syntans from the attempt in the early days to create products similar to vegetable tans down to today's commonly used simple syntans. Later he described the development of synthetic resins for the treatment of leather paying particular credit to the American developments. It is becoming more difficult to classify syntans on a common basis and it will probably be necessary to classify them according to their chemical structure.

Dr. G. Otto, head of the Leather Division of the Badische Aniline Und Soda Fabrik A. G. spoke of the electrical forces attracting and forming a combination of tanning product with animal skin. The ionic attraction and subsequent fixation are considered.

The writer presented a continuation of the series of papers dealing with the relationship of tannery processes under "The Inter-relation of Tannery Processes; part III". This covered raw stock, soaking and liming. The paper will be offered to the Journal of the American Leather Chemists' Association.

Dr. Burton wound up the Symposium with a resume of the work presented and drew attention to some of the new, recent work being pursued on the structure of collagen and the albuminoid fibers.

On Tuesday evening, a fine reception was given by the Stockholm City Council in the truly famous Golden Hall of the Town Hall of Stockholm. This building is one of the most magnificant in the world and is a tribute to Sweden, her architect, Ragnar Ostberg, and the towns people of Stockholm who contributed to funds for building this splendid monument to a country. We were welcomed by the Chairman of the City Council. For the Americans, it was a proud moment to observe in the murals depicting the evolution of mankind the Statue of Liberty among the gold statues of other landmarks of progress and freedom.

On Wednesday morning, a series of five papers were presented, the writer presiding. We call to your attention in particular the paper of Dr. E. Simoncini, Director of the Italian Leather Research Institute of Naples on "Some Lesser Known Properties of Leather". Simoncini emphasized that the adhesion of sole leathers to wet pavements is nill whereas it requires definite energy to move leather substitutes. The coefficient of friction of substitutes is greater than that for leather. The force required to perforate leather, for example to puncture by nails—is eight times that of the substitutes. The low thermal conductivity of leather is a controlling factor in foot comfort and is superior to substitutes. Dr. Kanagy gave a paper on a technique of using sonic, that is high velocity sound waves, to measure physical properties of leather by a non-destructive method. Such methods could be extremely valuable in studying changes in the physical properties of leather over long periods of time without destroying the samples. Dr. H. G. Wollenberg in a

joint paper with Mr. Holmes on "Tanning as a Rate Process", provided much interest in determining the rate or time it takes for hides to be fully tanned by a study of the "half period" since tanning is a dye-away or diminishing process. Dr. Mitton gave a paper on the "Diffusion of Water-Vapour in Leather" by using plugs of ground leather to minimize the different porosities of the grain, corium and flesh. The important result is that the transport of moisture takes place through the capillary spaces and not through the solid fibers. Mitton regards the ability of leather to pass moisture or water vapor as more important than the ability to transmit air.

In the afternoon the conference visited Skansen, the National Folk Park of Stockholm. Here a fine Swedish lunch was provided by the courtesy of the Swedish Leather Industry and the Allied Trades. In Skansen are preserved in an open park relics of the former trades and activities of Sweden. Particularly interesting to the group was an old tannery complete with lime pit, bark extractors, drying loft and even the living quarters of the tanner who was probably a prosperous citizen judged by the fact that over his bed was his tall beaver hat. The glass making exhibition was in full swing with glass workers creating many types of glass ornaments which were for sale. In the early evening, a boat ride was taken to the resort of Waxholm, on which many beautiful views of the surrounding sea and islands were seen with many noble houses in the distance. On the way back, the early evening twilight was illuminated by a brilliant moon which provided the making of many beautiful color photos by those on board.

Thursday, the final day of the conference, saw the reading of ten papers on a variety of topics important to leather making. Mr. E. B. Thorstensen of the American Leather Chemists' Association contributed to the discussion on the important problem of sewage or effluent in the leather industry which is becoming increasingly serious in Europe as it is in the United States.

The end of the afternoon was taken up with the final business of the conference. We received the reports of the Chairmen of the different commissions, transacted other business and arranged for the meeting of the next conference in 1957. The writer again welcomed the Union to visit the United States. As a result of other discussion, another committee on the fastness of dyes in leather was set up under Dr. W. Weber as provisional Chairman.

The writer regrets that because of official duties elsewhere, he cannot report on the results of the committee meetings but this was reported for the A. L. C. A. by our official delegates.

The final activity was a banquet in the Golden Hall of the Stadshus, the Town Hall, made available by the Stockholm City Council. This great and serene Golden Hall provided a fitting close to a truly fine and inspiring meeting. Mr. Moller, President of the Swedish Tanners Federation presided and gave a witty skillful speech in a mixture of Swedish, German, French and

English as a toast to the Union. Dr. Gustavson replied in a humorous vein reminding us that this wonderful town hall was once the site of a tannery and that we all had much to study in collagen. Dr. Swen Brohult, President of the Swedish Leather Chemists' Society spoke and was succeeded by Professor Grassmann, President of the German Leather Chemists' Society who extolled the merits and value of the connective tissues and their importance to life and health. A toast to the ladies was given by Dr. Mitton and called forth some amusing anecdotes both of European and American origin to which Mrs. Panikar De Pelache responded charmingly and graciously in several languages. As President Elect and representing the American Leather Chemists' Association, the writer spoke and expressed the hope that the Union would go on to greater importance from meeting to meeting and that the harmonious discussions of the many visitors from twenty-five different countries would augur well for a continuing harmony on other matters for the future. At two a. m. when the ball was over, the early sunrise of the northern latitudes began a new day, and we all felt that we had had a rewarding and rich experience in this meeting of leather scientists in Scandanavia.

The two delegates of the A. L. C. A. whose travelling expenses were paid for by the Association were C. Telander and J. Kanagy. Other members of the A. L. C. A. were:

Mr. E. Thorstensen (President-Elect) and Mrs. Thorstensen.

Mr. R. G. Weldon (Council) and Mrs. Weldon.

Mr. D. Meo (Council) and Mrs. Meo.

Mr. C. Martin and Mrs. Martin.

Mr. L. Bean and Mrs. Bean.

Mr. C. Haemiseggar and Mrs. Haemiseggar.

Mr. S. Dahl and Mrs. Dahl.

The following is the new slate of officers appointed by election by the delegates:

President, Dr. H. G. Turley, United States.

First Vice-President: Dr. G. A. Bravo, Italy.

Second Vice-President: Dr. K. H. Gustavson, Sweden.

Co-opted Members: Dr. P. W. Grassmann, West Germany.
Professor P. Chambard, France.

Honorary Treasurer: Dr. D. Burton, Great Britain.

Honorary Secretary: Mr. A. Harvey, Great Britain.

The next meeting will be held in Rome in 1957 with the Italian Leather Chemists' Society as host under the direction of Dr. Bravo.

The American Leather Chemists' Societies Association had a fine attendance this year of twenty-one members and relatives and we look forward to an even greater attendance in Rome. We urge as many of you as can to make the journey and meet our European colleagues in Rome. You will find much to learn, a different viewpoint and a greater stimulus from the professional contact with other scientists in the same field. You will also have interesting and rewarding experiences in the social way. Perhaps best of all is the experience of meeting interesting people who like you, have a sincere regard for leather and a continuing desire to study and improve leathers of the many types which are in use today.

REPORT ON THE MEETING OF THE INTERNATIONAL COMMISSION FOR LEATHER ANALYSIS

The Commission met four times during the Fourth Conference of the International Union of Leather Chemists' Societies held in Stockholm, Sweden, August 1–4, 1955.

Present at these four meetings were:

Dr. E. J. Baumann	(Switzerland)
Prof. Dr. E. Del Mastro	(Italy)
Mr. G. Forsyth	(England)
Prof. W. Grassmann	(Western Germany)
Dr. R. G. Mitton	(England)
Mr. Ir. J. Roelofs Heyrmans	(Holland)
Mr. C. G. Telander	(U. S. A.)
Prof. Weber	(Switzerland)
Prof. Ing. V. Dumoulin, *Chairman*	(Belgium)

The following matters were discussed and acted on:

1. An independent Commission for Physical Testing of Leather was established with Dr. R. G. Mitton as Chairman.
2. A Wiley type mill with 4 mm. screen was adopted for sample preparation.
3. The provisional method for determination of chrome by oxidation with perchloric acid after ashing was accepted as official.
4. Action on method for sampling of leather was postponed pending further study and suggestions by the membership.

5. As a result of previous International committee work, which amply illustrated the differences existing in the methods used in the various countries, it was decided to adopt as far as possible a general policy of using the average of all different conditions of analysis as a basis for further discussions. As a result, a drying temperature of 101 ± 1 °C. was adopted for all drying operations, leather moisture, oil determinations, and water soluble residues.

6. The following method for moisture determination was adopted: Weigh 2 g. of ground leather into a weighing bottle of approximately 50 mm. diameter and dry for three hours, reweighing at one hour intervals until weight loss does not exceed 2 mg. A study was proposed using pre-dried air.

7. Ash Determination. Three determinations were proposed:
   a. Water soluble ash. Evaporation and ashing of part of the water solubles solution at 600 ± 25 °C. and sulfating of the ash.
   b. Insoluble ash. Drying and ashing of the water extracted leather after determination of water solubles. This determination is necessary for the determination of combined tannin and degree of tannage.
   c. Total ash. To be determined by direct ashing or as the sum of the water soluble and water insoluble ash.

8. Oil. Two types of solvent have been chosen:
   a. Petrolic Ether, hexane.
   b. Chloroform, methylenechloride.
   Detailed method and apparatus will be arranged for. Objections were raised to the chlorinated solvents if the extracted leather is subsequently to be used for pH determination. A study of storage stability of chlorinated solvents using acid acceptor should be made.

9. Water Solubles Determination:
   Special tests will be run by the Commission members comparing the proposed "equilibrium extraction method" with the methods used by the individual members. The equilibrium method consists basically of extracting 10 g. of leather in a thermos bottle with 500 ml. of water, rotating gently by hand. Extraction time and temperature to be checked by a series of experiments in comparison with the Swiss and United States methods. By such a study, the Commission hopes to establish a condition of extraction which will yield an acceptable value to all members.

CARL G. TELANDER,
U. S. Delegate and Commision Member

Financing Waste Disposal Installations*

MR. CHAIRMAN, MEMBERS, AND GUESTS:

It is a real pleasure to me to meet with friends and colleagues of the Society after an absence of several years, and I count it a privilege to be able to make even a minor contribution to this meeting in Stockholm. I suppose, however, that I ought to be anxious about my topic for today, since I am not by profession a sanitary engineer or public administrator. In the second place, there is the matter of a title. The teachers of rhetoric recommend, I believe, that a title should predispose the audience favorably; yet my title makes not one but two unsavory references: sewage disposal and costs. But I am not really worried about that. To scientists there are no such things as improper or unacceptable facts; and the topic is of some importance to us all, not only as members of the leather industry but as members of the human race.

Most people in the civilized world, and certainly all in this audience, recognize the necessity of keeping our waters uncontaminated. Water is the one thing most needed to maintain life and health; it is a commodity nearly as important to industry as to agriculture. One conservation measure essential to maintaining clean water in adequate supply has been sewage disposal facilities for the processing of wastes. Sanitary engineers and chemists have devised installations by which these wastes may be safely returned to lakes, to streams, and to the earth. The question is no longer whether there is to be pollution control but rather who is to pay for it, and how much. My remarks today will be a report, necessarily brief, on how this question has been answered recently in the United States.

I say "recently," for even during the past few years much has been done in this field of public administration—to determine the costs of waste disposal and the distribution of these costs to construction, maintenance, improvement, and replacement.[1] This is not surprising, since nearly all American communities larger than 5000 people have sewage treatment facilities. Of course, if a factory is located in a very thinly populated area where community discharge is probably into a stream or lake, the industry must build its own disposal plant and meet certain requirements before its effluent may be emptied into the public waters. Such cases are clear: the industry bears the entire cost. But if the industrial effluent is to be processed in a municipal sewage plant, then the question of who pays, and how much, becomes complex. There is no single or simple answer, at least in the United States, for practice varies widely.

*Address delivered by E. B. Thorstensen, President Elect of The American Leather Chemists Association, Vice President, Research and Development, S. B. Foot Tanning Company, Red Wing, Minn., U.S.A., at the 4th Biennial Conference of the International Union of Leather Chemists Societies at Stockholm, Sweden.

There are many ways of financing sewage disposal plants, if all possible combinations were to be named. Two ways are an advalorem tax on property or special assessments on a front-foot basis. These are both inequitable, because, the amount of sewer service a property requires, like the amount of gas or electricity it requires, has little to do with the property value. A third and better way is to finance by special negotiated contracts on the "added cost" basis. This means that engineers estimate the costs of a combined system, then subtract the cost of a system without industry. This difference, or "added cost," is a good basis for setting up fair assessments and rates.[2] I know of one city where a packing company pays a fixed rental as well as a maintenance charge, another city where two packing houses pay 65 per cent of the construction and 90 per cent of the operating and maintenance costs of the treatment plant.[3] Where an industry is producing most of the objectionable sewage it seems reasonable for it to pay a large part of the construction and maintenance costs of a plant. A community plant to process the normal wastes of human habitation might be rather small. But much more equipment would be needed to process also dairy wastes or the heavy waste loads added by such industries as meat packing and rendering, metal and plating, food processing, oil refining, and by chemical industries, which would include tanneries, garages and gasoline stations, dyeing and drycleaning establishments, and breweries. The "added cost" system faces this fact in a very direct way.

The fourth way of financing sewage disposal is the sewage service charge. For my remaining time I shall talk about this, not only because it is more and more coming into favor but because it is so widely varied in plan. The American Public Works Association reported that in 1953 sewage service charges were in use in at least 581 communties—more than one fourth of the incorporated places over 5000 population—which indicates a nation-wide acceptance of such charges. One reason for this is that the sewage service charge places disposal facilities on a wholly or partially self-sustaining basis. The charges are for services rendered, and are not taxes or assessments. Revenues thus made available allows the undertaking of projects which would otherwise be impossible, because this type of charge allows the use of assessment and revenue type bonds. These issues can be excluded from tax limits and budget limit calculations. Thus communities can borrow funds to build disposal plants and to make other types of improvements and can, by properly calculated charges, allocate the costs of service equitably.[1]

Under such conditions, which differ from city to city, the annual per capita revenues vary tremendously—from a low of 64 cents to a high of $11.95 per capita, with an average of $3.30 for most communities smaller than 100,000. This wide variation results mainly from the variation in rate scales, which in each city are set up to meet a local pattern of costs. Rates in one city may

cover only the direct charges for sewage service; in another city they may have to provide amortization of sewage plant cost and to furnish sinking-fund contributions for eventual plant replacement. At least two other conditions influence cost. One is whether the sewerage system is separate or combined. The tendency today is to construct separate systems—that is, a storm sewer system for surface water and a sanitary system, which leads into the waste treatment plant. About two-thirds of American communities now have separate systems, which is in line with the current trend. The construction costs are higher, of course, but sewage treatment costs are lower. Another influence on costs is just how thorough the sewage processing is to be; the more thorough the treament required, the more expensive the plant is to build and to operate.[2]

Sewage service charges are based either on the volume of water consumed or on the constituents or amount of the sewage discharge. Industrial charges are usually based upon a percentage of the water bill. In all American communities the water waste disposal rates are lower than the water supply rates. But the picture is rather confused because these sewage rates may be based upon the number of employees in a factory, on the type of industry, on the size of the water meter, or on the number of sewer connections or the number of fixtures. Twenty-five cities between 5000 and 60,000 population use special, flat, or negotiated rates for industrial users,[1] and a number of cities operate on the kind of sliding scale used by Winnipeg, Canada. In Winnipeg the commercial rate per thousand gallons of water goes from 35 cents for small quantities down to 18 cents per thousand gallons for users of five million gallons or more per year. Sewer rental rates are related to this water consumption and go from six cents per thousand gallons for small users down to 4½ cents per thousand for the five-million-gallon users.[4]

More and more cities, however, are basing their sewage service charges not on the water bill but on the quantity or quality of wastes. The general principle is to make a normal charge for a normal load and an excess charge (surcharge) for a heavier load. Twenty-two American cities between 5000 and 50,000 people maintain a separate rate schedule for different quantities, and ten cities also consider quality of sewage. The following factors currently enter into such charges: (1) the constituents of the effluent; (2) the amounts to be considered "normal" and "excess"; (3) the rate scales; (5) the use of formulas; and (5) the frequency and methods of sampling. The major recurring constituents tested are the five-day B. O. D. at 20 degrees centigrade; the suspended solids; the hydrogen ion concentration; and the chlorine demand.

It is difficult to draw valid conclusions from such a diversified situation as I have described. It can be said, however, that the true importance of sewage disposal is rapidly becoming recognized by public administrators and by

industry, and that in the United States the sewage service charge, adapted to local conditions, is coming to the fore as the best method for most communities to handle fairly the problem of sewage disposal costs.

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Note On The Chemistry Of Black Wattle Tannins

Acetyl Values, Criteria of Purity and Molecular Weights

By D. G. ROUX and S. R. EVELYN

The conflicting views on the structure of wattle and other vegetable tannins put forward by Putnam and his coworkers [1,-19] on the one hand and by the Forestal workers [10, 11, 12] and by one of us [13, 14, 15] on the other has led to much confusion in the minds of leather chemists. For example, Lollar [16] has concluded, in a recent review of tannin chemistry, than an "impasse" exists between the findings of Putnam on the one hand and those of White and co-workers [10, 11, 12] on the other. The chief, though not only point of difference is the validity of the method of hydrolysis used in the determination of acetyl values [13, 1].

Dr. Putnam has kindly made available to us a sample of his purified wattle tannin and so has made possible a re-examination of this question by means of a more direct comparison between his and our starting materials. This paper represents the results of this re-examination in three sections, viz. investigation of the purity of samples, determination of number-average molecular weight, and determination of acetyl values.

a. Criteria of Purity

Putnam and Gensler [4] purified wattle tannin, by partitioning the commercial extract (60 gm.) in a water, ethanol, acetone, ethyl acetate (2:1:3:12) mixture. The organic phase was evaporated to dryness, the residue repeatedly redissolved in methyl ethyl ketone and finally precipitated from such a solution with ether. An authentic sample of the product, which Putnam and Bowles [8] later claimed to be homogeneous from its chromatographic behaviour, was kindly supplied by Dr. Putnam for examination.

A one-dimensional chromatogram of this sample together with the reference-compounds fructose, glucose and sucrose was run for 18 hours in butanol/acetic acid/water. (See Roux[14]). After spraying with naphthoresorcinol-phosphoric acid reagent[27] and heating at 80°C. the developed chromatogram showed that fumes which remain at the origin, were either absent or else present in low concentration. The sugars, sucrose, glucose, and fructose present in the commercial extract[14] were prominently evident in Putnam and Bowles "purified" tannins. (Fig. I).

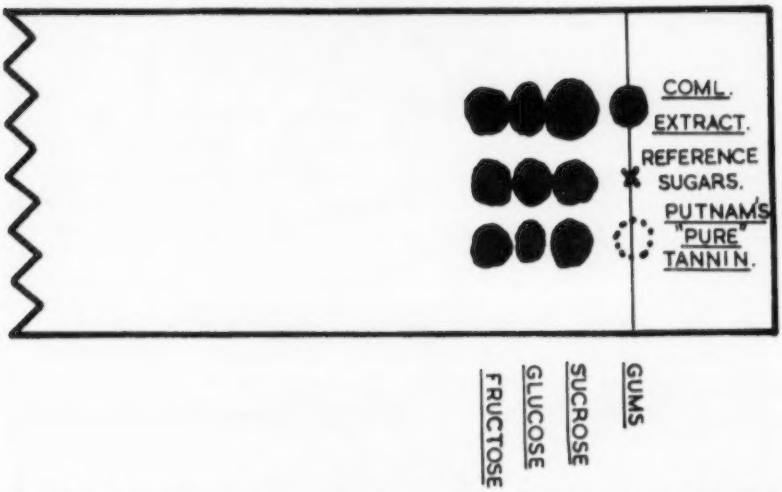

FIGURE I.—One dimensional Chromatogram of Black Wattle Extract and Putnam's Purified Tannin. Irrigant: Butanol-Acetic Acid - Water (4:1:5) sprayed with napthoresorcinol-phosphoric acid mixture and heated at 80°C. Sucrose and fructose spots were pink, and glucose blue in colour.

Two-dimensional chromatography of Putnam's sample using water-saturated sec.-butanol for direction 1 and water-saturated phenol or water only for direction 2[15,28] followed by spraying with ammoniacal silver nitrate, showed the presence of a number of strongly-reducing tannin constituents (Fig. II). The constituents present, A, B, C, D, and E (plus an intense trail) are typical of the polyphenolic mixture present in the unoxidised fresh-bark extract . (Fig. III). Constituents F, G and H, were either absent or present in very low concentration. The RF values of the constituents A-E were the same in both solvent systems in Figs. II and III.

Putnam's sample of "pure" tannin was light pink in color, and in the dried condition remained stable indefinitely. This is reflected by the fact that mobile constituents A-E (Fig. II) corresponded in RF value with those in the fresh-bark (Fig. III). and commercial extracts.

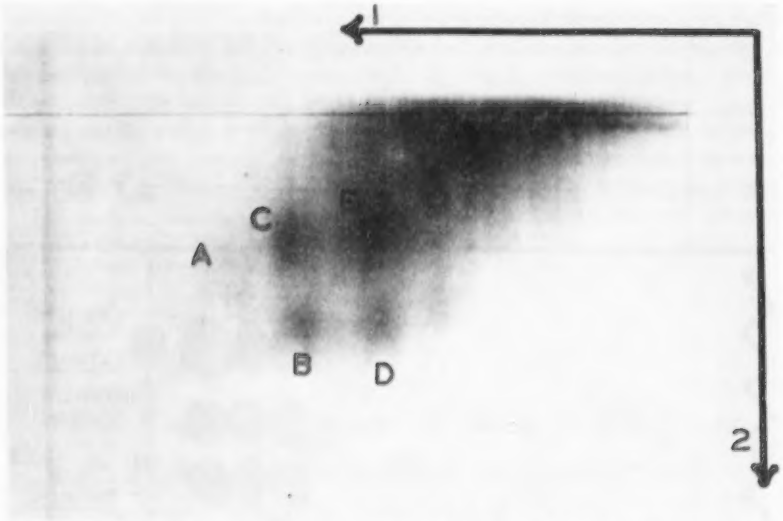

FIGURE II.—Two-dimensional Chromatogram of Putnam's "Purified" Wattle Tannin Sample. Sprayed with ammoniacal silver nitrate.
Direction 1: Water-saturated sec.-butanol
Direction 2: Water

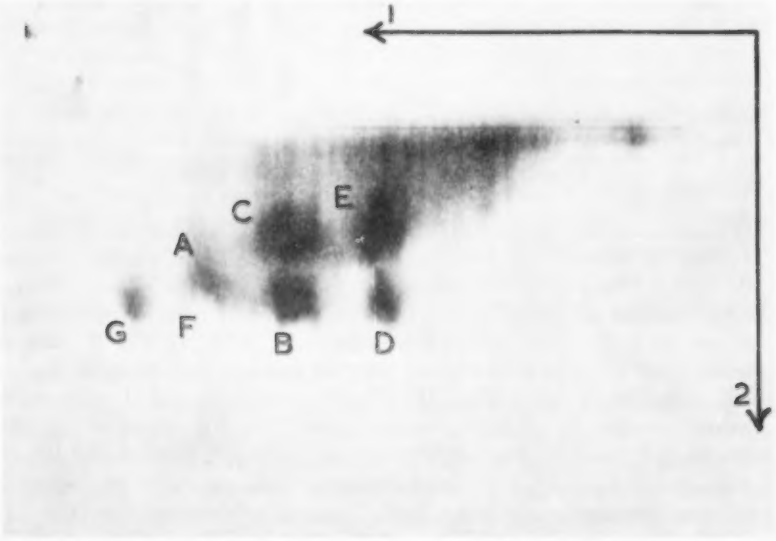

FIGURE III.—Two-dimensional Chromatogram of Black Wattle Fresh Bark Extract. Spray and irrigant as for Figure II.

This chromatographic evidence shows that:

1. Putnam and Bowles' "pure" tannin sample contains simple sugars, which will acetylate with the tannins. The tannin sample is, therefore, not "pure".
2. The tannins present in their "pure" sample consist of a mixture of phenolic constituents, and Putnam's single formula for wattle tannins, $C_{30}H_{26}O_{12}$ is not tenable.
3. Putnam and Bowles'[8] solvent system selected for chromatography of the polyphenols (tertiary butyl alcohol and 0.1*N* (pH = 9.0) buffer solution 1:1) gives no resolution of the tannin constituents.
4. The high RF polyphenols present in their tannin sample have the same properties as unoxidised (colorless) polyphenols derived from fresh black wattle bark, in partioning solvents (direction 1) and under conditions of absorption (direction 2) on cellulose sheets. Their sample appears to have undergone no major change in transit or storage.

(b) Number-Average Molecular Weights

A modified Ray[29] ebulliometric method of molecular weight estimation for diazomethane methylated wattle tannins, freed from gums and sugars has been developed at this Institute[28, 30, 31]. This method, using benzene as solvent, was applied to the "purified" tannin sample submitted by Dr. Putnam.

EXPERIMENTAL:

0.3832 gm. tannin sample was dissolved completely in 30 ml. methanol, and methylated with excess diazomethane. The usual procedure[31] gave a white powder, and after drying for 2 hours in an Abderhalden pistol at 110°C. over P_2O_5, the yield of methylated product was 0.3582 gm.

Methoxyl content = 31.22 and 32.11 per cent

Yield: Wt. tans in methylated material.

$= 0.3582 \times .8570^{*} = 0.3070$ gm.

Wt. of Tans taken $= \dfrac{0.3822 \times \frac{88^{**}}{100}}{} = 0.3372$ gm.

Yield = 91.0 per cent (not accounting for mechanical losses in manipulation).

*Conversion factor[30] for converting molecular weight of methylated tannins into that of tannins.

**Assuming 88 per cent purity obtained by Buchanan *et al.*[32] by this purification procedure.

Number-Average Molecular Weight in Benzene:

Values:	$\frac{\text{Deflection in mms.}}{\text{Concn. in gm/100 gm.}} = \frac{\triangle D}{C}$
	279.90
	272.60
	266.48
	274.05
Mean	273.26

Molecular weight of diazomethane-methylated tannins = 1483 ± 15
Molecular weight of original tannins = 1270 ± 13

Putnam and Gensler[4] by comparison using the Rast method with camphor as solvent, obtained a value of 926 for an acetylated product, isolated in 30.5 per cent yield. On the basis of their own estimation of acetyl content (48 per cent) this corresponds to a molecular weight of 0.5312 x 926 = 492 for the tannin itself. Their formula $C_{20}H_{26}O_{12}$ corresponds to a value of M = 578.5. Molecular weight estimations on the acetylated tannins is in any event unsatisfactory as the sugars and gums present with the tannins are also substituted during acetylation and, therefore, remain as impurities.[31, 33].

Due to the well-known fact that benzene, camphor (see Robinson and Robinson[34]) and almost all solvents give anomalous molecular weight values at times, Roux[28] recommended molecular weight comparisons in various solvent systems by various methods. Comparison in bromoform in particular was recommended, as the cryoscopic method with bromoform was shown by Klages et al[35] to give normal molecular weights for the methoxyl and acetyl derivatives of ten synthetic polydepsides. Evelyn[30] in following up this suggestion obtained excellent agreement between the ebulliometric (benzene as solvent) and cryoscopic (bromoform as solvent) methods for both methylated and acetylated wattle tannins. The above molecular weight (1270 ± 13) of Putnam's tannin sample, therefore, excludes the possibility of an anomalous value due to association of the methylated tannins in benzene.

Evelyn[31] obtained number-average molecular weight values very close to 1270 for wattle tannins from a number of sources, and Putnam's values for the acetylated product as well as the calculated value for his formula are less than half the actual value. This evidence alone, invalidates his proposed single formula, $C_{20}H_{28}O_{12}$, for wattle tannins.

(*c*) *Acetyl Values*

Putnam and Bowles[1] in the reply to criticism by one of us[13] report the following findings:

1. Unsubstituted tannins show negligible keto-absorption when examined by infra-red spectroscopy.

2. Unsubstituted tannins give significant acetyl values (3.9-8.8 per cent) by the alkaline hydrolysis method.

3. The trans-esterification method as used by them on empirically purified (see Criteria of Purity) tannins, gives low but appreciable acetyl values (0.9 – 3.3 per cent).

4. The alkaline hydrolysis method gives a much higher acetyl content (48.01 per cent) than the trans-esterification technique (33.08 per cent).

Roux[17] independently established that methylated wattle tannins (36.5 per cent methoxyl) exhibit no keto-absorption in the infra-red spectrum. Putnam and Bowles finding under 2) is however in complete conflict with the infra-red evidence. In certain exceptional cases the trans-esterification method is known to furnish low acetyl values in the complete absence of such groupings[18], and finding 3) is therefore probably correct. Statement 4) supports Roux's contention[13] that differences in acetyl values reported by himself and by Putnam et al., are *not* due to Putnam's claim of a higher degree of substitution (so-called "peracetylation"), but are probably due to differences in the methods used for acetyl estimation.

More recently Putnam et al.[3-6] claimed the removal of "acetyl impurity" (see 2) above) present in both quebracho and wattle tannin samples, previously used as basis for his investigations. It should be noted that he produced the same effect with "commercial" catechin[5] (7.87 per cent acetyl), which was reduced to zero acetyl content on "special purification"[1]. The empirically purified tannins could again be "peracetylated" (45.5 per cent and 47 per cent acetyl for quebracho and wattle respectively), and furthermore agreement was claimed between the alkaline hydrolysis technique and a radioactive method of acetyl estimation on incompletely substituted quebracho (25–26 per cent acetyl). The latter evidence was produced to prove that "peracetylation" is, in fact, possible at their hands.

Nevertheless, proof that Putnam's acetyl values for both the tannins and their acetylated derivatives are incorrect, and that the alkaline hydrolysis technique as applied by Putnam and co-workers cannot therefore be used for wattle and other tannins is furnished below.

Choice of Method for Acetyl Determination

The problem of selecting a suitable method of acetyl estimation for a particular class of compounds is not new (see Houben-Weyl "Methoden der Organischer Chemie Band II. Analytische Methoden." G. Thieme Verlag, Stuttgart, 1953. p. 343). Certain classes of compounds are known to form acids as a result of acid or alkaline hydrolysis, and Freudenberg and Harder [18], who developed the acid hydrolysis technique using p-toluene-sulphonic acid, suggest that tannins and sugars should be tested first for their tendency to form volatile degradation-products with acid or alkali. For tetra-acetyl-dl-epiquebracho-catechin Freudenberg and Mailand [19], for example, found that alkaline hydrolysis gave abnormally high values, probably due to the formation of volatile acids. On the other hand chromic acid digestion [20] *which completely oxidizes the C_{15} skeleton without attacking the acetyl groups*, gave satisfactory results.

Robinson and Robinson [34] found that an acetylated leuco-anthocyanidin, peltogynol, gave high acetyl values when using the method of alkaline hydrolysis. This was due to a small porportion of volatile acid produced by the degradation of the compound itself. Similarly for wattle tannins, evidence provided by Putnam (e. g. relatively high acetyl values for unsubstituted tannins known to show negligible keto-absorption), and by others (see [13]), clearly point to the formation of volatile acids during the alkaline hydrolysis method of acetyl estimation.

As for epiquebracho-catechin, further proof for wattle tannins may be provided using the Kuhn and L'Orsa [20] chromic acid method, and the Kuhn-Roth [21] apparatus for completely oxidizing the tannin structures without affecting the liberated acetyl groups.

Experimental:

Apparent Acetyl Content of Wattle Tannins:

The Kuhn-L'Orsa [20] method was used for the following estimations:-

Lead-salt purified [22] black and wattle polyphenols from the commercial extract (88 per cent recovery) gave a value of 1.03 per cent acetyl.

Fully methylated (-OCH_3 – 36.5 per cent) wattle polyphenols, obtained by methylating the lead-salt purified tannins with dimethyl sulphate gave a value 0.78 per cent acetyl groups.

Acetylated Wattle Tannins:

Fully acetylated wattle tannins (acetic anhydride + sodium acetate) were examined by both trans-esterification [21] and the Kuhn-L'Orsa [20] chromic acid methods:

Found:

Method	Per cent Acetyl	
Trans-esterification	37.8	38.0
Chromic Acid	37.9	38.2

Discussion of Results:

The above results clearly show that negligible acetyl values (0.06 – 0.08 acetyl groups/C_{15} unit) are obtained from unacetylated wattle tannins by the chromic acid method without elimination of "acetyl impurity" claimed by Putnam and co-workers[3,6]. This is in agreement with the negligible keto-absorption obtained from infra-red spectral examination of methylated black wattle tannins, found by both the author and Putnam.

For the fully acetylated wattle tannins the chromic acid and trans-esterification methods are in close agreement. This contrasts with Putnam's finding[1] that the trans-esterification method gives substantially lower values (ca. 15 per cent) than the alkaline hydrolysis method for "per-acetylated" and deacetylated quebracho, wattle and mangrove extracts.

No explanation is offered for the recently-claimed concordance between radio-active and alkaline hydrolysis methods by Putnam and co-workers. Putnam subsequently found himself unable to supply a sample of any of his peracetylated tannins to the Authors for analysis by the chromic acid method.

Nevertheless, evidence forthcoming from numerous reliable sources (see above and [13]) show that the alkaline method of acetyl estimation is capable of erroneously high values for a variety of polyphenolic compounds. For unacetylated wattle tannins the chromic acid method reflects very low acetyl content (1 per cent), whereas Putnam found 3.87 per cent acetyl by the alkaline method[1]. Also for deacetylated (previously "peracetylated") tannins Putnam[1] gives the values: trans-esterification = 0.62 per cent acetyl; acid hydrolysis = 0.99 per cent acetyl and alkaline hydrolysis = 18.42 per cent acetyl. The above values and Putnam's abnormally high value of 48 per cent acetyl for his "peracetylated" wattle tannin by the alkaline method of hydrolysis, as used by Putnam and co-workers, are probably due to the formation of volatile degradation products. Similar criticism of Putnam's abnormally high acetyl values was made by White and co-workers [24, 25] for quebracho polyphenols.

For black wattle polyphenols the chromic acid digestion method reaffirms Roux's[26,17] previous finding that a high proportion of ether linkages are present in the tannins (ca. 1.8 – 1.9 per C_{15} unit).

Summary

Some aspects of the work of Putnam and co-workers on wattle polyphenols (tannins) are critically examined.

Paper chromatography is used to show that Putnam and Gensler's "purified" wattle polyphenols (tannins), as supplied by them, contains sugars but no gums. Two-dimensional paper chromatography shows that a similar mixture of polyphenolic compounds or tannins is present, as was previously found in wattle fresh-bark extract. Putnam's single formula for wattle tannins is, therefore, untenable.

Accurate molecular weight estimations, in which the possibility of anomalous values is excluded, show that the polyphenols present in the same sample have an average molecular weight of 1270 ± 13. This value is more than double that of Putnam's $C_{30}H_{26}O_{12}$ formula (M.W. = 578.5), as well as that calculated from his molecular weight determination on fully acetylated tannins.

Proof is provided that the alkaline hydrolysis technique, as applied by Putnam, gives erroneously high acetyl values on fully acetylated black wattle wattle. "Peracetylation" claimed by Putnam does not occur, and in the known absence of keto groups this reaffirms the presence of a high proportion of ether linkages in wattle polyphenols previously claimed by Roux.

All the above findings suggest that complete revision of the work by Putnam and co-workers is necessary.

Acknowledgment

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Resistance of Purified Collagen to Degradation by Salt-Tolerant Bacteria in Pure Culture*

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Skins from many kinds of animals may be converted into durable leather products of a thousand uses. Freshly removed skins are not considered a suitable raw material for the tanner because they are so highly perishable. Unavoidable delays between time of slaughter and the beginning of processing make it necessary to preserve hides and skins immediately after removal from the animal. Spoilage may be prevented by drying the skins, but in most countries it is customary to use a curing process employing strong solutions

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of sodium chloride. Salt, however, is not an ideal preservative. Under adverse conditions, and especially during hot weather, bacterial damage may occur and cause a loss of collagen, the actual leather-making substance of skin. This paper presents a study of specific collagen degradation at high levels of salt, employing pure culture conditions with bacteria isolated from hide-curing environments.

Curing may be done slowly, between layers of dry salt in a pack, or more rapidly by soaking in saturated brine. The primary function of the salt is to inhibit bacterial growth by a combination of effects such as dehydration, toxic action, enzyme inactivation, etc. Temperature and relative humidity are important factors. A secondary action is the partial removal of globular proteins from skin which not only encourage bacterial growth but also are undesirable for leather-making purposes.

After salt curing various forms of bacterial damage have commonly been observed. Most noticeable of these are the red or violet flesh stains, especially the condition described as "red heat". The organism responsible for such action are halophilic and are known to originate mainly from marine salt [11, 18]. Other types of bacteria normally present on the animal's skin at the time of slaughter may be of soil or intestinal origin. Those that possess salt tolerance and proteolytic activity may cause destruction of the skin substance or grain surface. Such organisms may be quite adaptable [1], enabling them to grow under changing concentrations of salt and moisture. Some interesting concepts to explain this behavior have recently been explored [16]. Chemical additives have been recommended [10, 21] to improve the preservative action of salt; or the numbers of bacteria may be greatly reduced by thoroughly washing the hides before curing [6].

Chemical methods for evaluating the extent of bacterial damage to hide proteins have usually been limited to measuring the various forms of soluble nitrogen liberated. Volatile nitrogen in particular has been advocated as the best criterion of curing conditions [9, 20]. However, since none of these methods was specific for collagen itself, they gave only indirect evidence of its degradation. Mammalian collagen, or the gelatin derived from it, is the only common protein containing a large amount of the amino acid hydroxyproline (13-14 per cent). Determination of this compound by the colorimetric procedure of Neuman and Logan [14] offers a specific method for estimating collagen in soluble or insoluble form.

A prime necessity for pure culture studies is a suitable sterilization method. This was a stumbling block for many years because of the extreme heat sensitivity of collagen. Finally, Maxwell [12] developed a chemical method for use with sheepskin based on the action of sulfurous acid. After modification as suggested by Anderson [2] to prevent swelling of the fibers, this has

proven to be a very satisfactory treatment. There was no apparent effect on the properties of collagen.

Materials and Methods

Culture media. For isolating and maintaining cultures, nutrient agar (Difco) was enriched with yeast extract 0.6 per cent, tryptone 0.2 per cent, glucose 0.2 per cent and salt 12 to 23 per cent as indicated. For the screening and collagen tests a broth was prepared by dissolving the yeast, tryptone, glucose and salt in water*. A study of growth factor requirements by Katznelson and Lochhead [5] emphasized the value of yeast extract for stimulating growth.

Isolation and screening of cultures. Samples of used salt, brine and salted hide were obtained from commercial slaughterhouses; laboratory curing experiments and pieces of fresh hide also served as source materials. Salt-agar dilution plates prepared from these samples yielded a large collection of salt tolerant and halophilic bacteria. Duplicate plates without salt were used to isolate another group for adaptation experiments. Although most of the organisms were facultative, no obligate anaerobes could be found. After purification all cultures were screened for ability to liquefy 12 per cent gelatin. It is generally accepted that "collagenase" activity implies a similar action against gelatin [4]. With this assumption it was possible to eliminate the totally inactive isolates from further testing. Some of the cultures were screened also with commercial hide powder, but the results obtained were identical to those with gelatin. This confirms the experience of Grassmann [7] and many others concerning the denatured character of the collagen in hide powder.

As a result of screening a group of 67 salt tolerant cultures was selected for the collagen tests. No attempt was made to classify them beyond examination of stained preparations. Anderson [3] recently reviewed the literature concerning bacteria involved in red discolorations. Some of the cultures in our collection (groups D and E below) were of this type. They are notable for being true halophiles and sensitive to water or dilute salt solution. Other workers in the field have placed their isolates in recognized classes or genera [1,9]. But for our purpose, identification of the cultures was of no immediate concern.

Adaptation. Another group of 106 cultures isolated on salt-free media was adapted to growth with salt by repeated transfer to tubes of broth containing gradually increasing amounts of salt. A total of 75 strains attained good growth in 2 M salt (ca 12 per cent) and a number of these could also tolerate 2.5 M. However, the 3M level proved to be a critical point beyond

*Ingredients such as peptone and gelatin were undesirable in the test medium because of their hydroxyproline content.

which none of the adapted strains would grow. This behavior seems interesting in view of the fact that most of the cultures originated from saturated salt environments (above 5N). Since the majority were spore-forming rods it is probable that these types survived in the form of spores.

Collagens. Cowhide collagen was used as substrate for testing activity of the organisms. Only the center layer of hide was utilized since this is probably the purest natural form of collagen. One preparation had been treated according to commercial practice for limed hide; i. e., it was salt-cured, soaked in water and then in saturated lime liquor long enough to remove the hair. After drying and cutting into small pieces this was designated as "limed collagen". The other preparation was "unlimed collagen", treated mildly to avoid denaturation so that it resembled what is often referred to as "native" or purified collagen. It was extracted with salt solution, dilute lime water and solvent to remove practically all impurities.* The two preparations were directly compared in the tests, and differences may be attributed mainly to the action of lime.

Sterilization. Sterilization of collagen was accomplished essentially by the method of Maxwell[12]. Collagen pieces (5–10 g. lots) were treated in succession, in closed containers, with 100 ml. of each of the following solutions: (1) 0.025 M. sodium metabisulfite for 17 hr.; (2) 0.3 M hydrogen peroxide for 7 hr.; (3) sterile 0.1 M sodium bicarbonate for 17 hr.; (4) two changes of sterile 10 per cent salt. The bisulfite soln. (4.75 g/L) was adjusted to pH 2 with strong HCl before making to volume. The first three solutions also contained 10 per cent added salt to prevent swelling. After the stated times, each reagent was decanted and replaced by the next in the series. Finally, the treated pieces were transferred aseptically to tubes of sterile salt broth to form the complete medium. Anderson[2] recommends treating in buffer soln. after sterilization, but this was not found to be necessary, since final pH was about 6.8–7.0.

Test Procedure. Tubes of the complete medium, each containing 1 or 2 pieces of sterile collagen, were incubated for several weeks at 30°C. to check sterility and then inoculated with the test cultures. Incubation was continued in a moist chamber for 2 to 3 months before subjecting the materials to chemical analysis. Stuart[20] and others have emphasized the long lag period and slow growth rate characteristic of halophilic bacteria, which require extended incubation for proper evaluation of their activities.

In this study, degradation of collagen was measured by determining the relative amount converted to soluble form, as estimated by corresponding amounts of hydroxyproline. Soluble matter was separated by centrifugation, and the residues were washed once with warm water. Aliquots of the combined

*This was a portion of sample 19 for amide nitrogen analysis by Mellon, Viola, and Hoover[13] of this laboratory, to whom we are indebted.

supernatants were used to prepare acid hydrolysates of the soluble fractions, by autoclaving with 6 *N* HCl in sealed tubes (20 lb. – 4 hr.). Insoluble residues were decomposed with warm acid, transferred to small tubes and hydrolyzed in the same manner.

Hydroxyproline was determined colorimetrically in the neutralized hydrolysates by the Neuman and Logan method [14].

$$\text{Per cent activity} = \frac{\text{mg. soluble hydroxyproline}}{\text{mg. total hydroxyproline}} \times 100,$$ where total hydroxyproline is merely the sum of the soluble and insoluble fractions. The range of values for uninoculated controls is included in the tables to show the extent of non-bacterial effects.

Results

Halophilic organisms. Single cultures of the 67 strains were first tested on both limed and unlimed collagen. Some representative results are shown in Table I. The organisms are grouped according to gram stain and mor-

TABLE I

Salt Tolerance and Collagen Solubilizing Activity of Representative Single Cultures

Cultures	Growth without Salt	Growth with 4M Salt (23%)	Activity* in 15% Salt Broth + Substrate		
			12% Gelatin	Limed** Collagen	Unlimed*** Collagen
Uninoculated controls	—	—	0	10–20%	3–9%
Gram-pos. rods:					
No. 12	Yes	No	+++	62	8
No. 11	Yes	Yes	+++	32	5
No. 87	No	Yes	++	34	6
Gram-neg. rods:					
No. 21	Yes	Yes	+++	41	5
No. 22	Yes	Yes	+++	36	3
No. 20	No	Yes	++	20	4
Gram-pos. cocci:					
No. 28	No	Yes	++	98	5
No. 78	No	Yes	++	31	7
No. 59	Yes	Yes	+	25	7
Gram-var. red cocci:					
No. 112	No	Yes	+	20	6
Gram. var. irreg. rods:					
No. 104	No	Yes	++	20	10

*Gelatin, complete liquefaction: +++in 4 weeks; ++ 4 to 8 weeks; + over 8 weeks. Collagen, solubilization: hydroxyproline in solution as per cent of total found.

**Center layer of cowhide soaked in saturated lime liquor; chemically sterilized.

***Center layer of cowhide carefully purified without liming; chemically sterilized.

phology, with notations in the next columns on the requirement for salt. It might be added that many of them will tolerate complete saturation, notably those requiring salt for growth. Uninoculated controls showed an appreciable amount of degradation of limed collagen, but none with the unlimed. Two of the cultures, No. 12 and No. 28, were highly active against limed collagen, and a number of others were moderately active. But none of the cultures showed any significant activity with unlimed collagen.

Table II gives some of the results of experiments with mixed cultures. The members of each morphological group were pooled and tested as group

TABLE II

Enhanced Activity Shown by Various Mixed Cultures

Composition of Culture Mixtures	Total No. of strains	Activity* in 15% Salt Broth + Substrate	
		Limed** Collagen	Unlimed*** Collagen
Uninoculated controls	0	10–20%	3–9%
A Gram-pos. rods	19	47	10
B Gram-neg. rods	5	32	5
C Gram-pos. cocci	30	40	5
D Gram-var. red cocci	10	15	8
E Gram-var. irreg. rods	3	42	8
Group Combinations			
A + B	24	65	8
A + C	49	29	—
A + D	29	22	—
A + E	22	41	11
B + C	35	37	5
B + D	15	21	6
B + E	18	91	11
C + D	40	24	—
C + E	33	62	14
D + E	13	58	8
A + B + C + D + E	67	93	15
Selected Mixtures			
No. 19 (B) + E	4	88	36
No. 104 (E) + B	6	93	40

*Collagen solubilization: hydroxyproline in solution as per cent of total found.
**Center layer of cowhide soaked in saturated lime liquor; chemically sterilized.
***Center layer of cowhide carefully purified without liming; chemically sterilized.

mixtures. Again there was moderate activity against limed collagen, but no definite evidence of enhanced or synergistic effect. And again there was no action on the unlimed collagen. Then the group mixtures were tested in all possible combinations with each other. A complete range of values was obtained, but lack of space prevents showing them all. The grand mixture of all

5 groups was consistently highest, averaging above 90 per cent activity on limed collagen but with insignificant action on the unlimed. In studying the results it was concluded that combinations containing group B, group E or both were the most active. Consequently further mixtures were prepared to narrow the effect. Two examples shown at the bottom of the table illustrate the enhanced activity of these mixtures against both substrates. This was the only occasion where we ever detected significant degradation of unlimed collagen in a salt medium.

Adapted organisms. As described above, a group of 75 cultures from salt-free media had been adapted to grow in the presence of 12 per cent salt. Table III lists some representative results when these organisms were tested before

TABLE III

Influence of Salt Content on Activity of Salt-adapted Cultures

Cultures	Activity* in Broth with and without Salt + Substrate				
	Hide Powder No Salt	Limed** Collagen No Salt	Limed** Collagen 12% Salt	Unlimed*** Collagen No Salt	Unlimed*** Collagen 12% Salt
Uninoculated controls	0	4–14%	12–22%	1–2%	1–2%
Gram-pos. sporing rods					
No. 23	+++	98	87	12	5
No. 42	+++	100	46	20	4
No. 67	++	99	50	19	3
No. 69	++	99	22	32	3
No. 77	+++	100	17	57	3
Gram-pos. nonsporing rods					
No. 2	++	98	32	5	3
No. 3	++	98	29	12	3
No. 5	++	77	24	9	4
No. 10	++	97	29	10	4
Gram-pos. cocci					
No. 102	+	13	50	2	2
No. 106	+	27	13	8	2
Gram-neg. rods					
No. 105	++	37	59	2	2

*Hide powder, complete solubilization: +++ in 4 weeks; ++ 4 to 8 weeks; + over 8 weeks. Collagens, solubilization: Hydroxyproline in solution as per cent of total found.

**Center layer of cowhide soaked in saturated lime liquor; chemically sterilized.

***Center layer of cowhide carefully purified without liming; chemically sterilized.

and after adaptation. Without salt most of these cultures were highly active against limed collagen. Also a number of them showed some action on the unlimed collagen in the absence of salt. However, when tested with 12 per cent salt after adaptation, activity sharply declined in almost every case. The important point is that all activity against unlimed collagen had disappeared.

Discussion

The results obtained indicate that purified collagen, largely unaltered by chemical or physical change, is quite resistant to bacterial attack in high salt media. Some members of the genus *Clostridium* have been shown to possess collagenase activity [1,5], as well as the enzymes papain [5] and pepsin [1,7]. But such agents would not be expected to operate in the presence of high salt concentration. Since curing damage does occur, however, other organisms may be a source of "collagenase", especially when present in suitable combinations and under favorable conditions. A more practical finding is confirmation of the importance of avoiding contact with denaturing effects such as moist heat and alkali.

Summary

Salt-tolerant and halophilic bacteria were isolated from salted hides. Those cultures able to liquefy gelatin were tested for solubilizing activity on limed and unlimed collagen. A modified chemical method was used to sterilize the collagen pieces without apparent alteration of properties. Bacterial degradation was measured by colorimetric estimation of the proportion of hydroxyproline in soluble form after suitable incubation.

Results were obtained for single cultures as well as various combinations of pooled mixtures. In general the unlimed or "native" collagen proved extremely resistant to attack, while many of the strains were moderately active on the limed material. In only a few cases, selected culture mixtures appeared to degrade both forms of collagen. Other tests with organisms adapted to salt tolerance showed the flexible nature of this adaptive property, and the effect of salt on their collagen-solubilizing activity. It was concluded that synergistic activity may well account for bacterial damage under curing conditions, and that alkaline denaturation greatly increases the susceptibility of collagen to bacterial attack.

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Physical Properties of Fatliquored and of Solvent Extracted Leathers*

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Abstract

Past experiments made on the influence of fatliquoring on the physical properties of leather can be questioned from the standpoint of statistical principles. As a basis for studying and clarifying the influence oil has on the physical properties of leather, statistically designed experiments were carried out comparing the physical properties of fatliquoring leather to solvent extracted leathers. Such physical tests as tensile strength, stitch tear strength, tongue tear strength, ball burst strength and hystersis curves were conducted and the data analyzed. A discussion of the results is presented and emphasis placed on those tests that best distinguish what the fatliquor does to the leather.

While various aspects of the fatliquoring problem have been studied extensively in the past it is only in recent years that the influence of fatliquoring on the physical properties of leather has been studied. In a previous article by Hamel and Roddy[1] their literature review indicated the status of the physical tests they performed in studying fatliquoring. They stated that

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statistically planned experiments had been carried out in only a few of the investigations. In 1953, Zacharias, Mann, Steiner and Horkowitz [3] published their statistical studies on the influence of grease content and fatliquoring versus stuffing procedures as they influence the physical properties of variously tanned side upper leathers. Schuttleworth [2] in the same year published his studies on the influence of oil on the physical properties of leather and, based on a statistical analysis of the data obtained, concluded that some of the previous work should be considered questionable as to the validity on strength characteristics.

As a basis for studying and clarifying the influence fatliquor oils have on the physical properties of leather statistically designed experiments were carried out comparing the physical properties of fatliquored leather to solvent extracted chrome in the blue stock which had received no fatliquor. Such physical tests as tensile strength, per cent stretch at break, stitch tear, tongue tear, ball burst strength and hysteresis curves were conducted and the data analyzed. A discussion of the results is presented from the standpoint of those tests that best distinguish what the fatliquor does to the leather.

Experimental

The decision was made to select treatments that would exhibit differences in degree of cohesive forces between the fibers and differences in the physical properties of the leather. The treatments were air drying some of the non fatliquored stock to produce stiff leather, fatliquored leather which gave a soft, pliable leather, and solvent treated stock giving a leather which exhibited softness and pliability without the presence of oils.

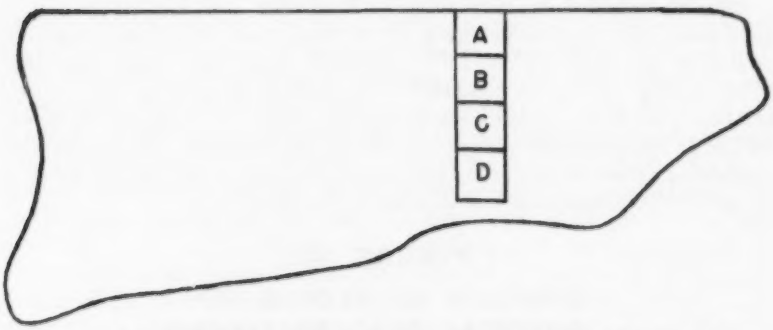
After selecting the desired treatments, the next problem was the selection of a representative stock sample. Considering the variability of the physical properties of leather, between skins and within skins, it was apparent that the best sample to use for a study of this kind would be whole skins. Processing whole skins in the laboratory presented the problem of obtaining uniformity of stock and sufficient skins on any one run to make the results worthwhile. Therefore, to obtain a representative sample a tanner agreed to give us shoulder strips from kip skins of chrome in the blue.

Two separate lots, each containing twelve shoulder strips of commercially prepared chrome tanned kip skins in the blue were used in these experiments. The strips were cut from the shoulder ends of sides after the shoulder had been cropped as shown in Figure I. The strips were further subdivided into 5 by 5 inch blocks as shown in Figure I. The lots of twelve strips each were designated as Lot I and Lot II respectively. The reason for using 5 by 5

inch blocks was based on past experience, the length of the shoulder strips, and the number of physical test samples to be cut from each block.

FIGURE 1

STRIPS OF CHROME TANNED KIPSKINS USED (APPROXIMATELY 5" BY 25")



THE BELLY EDGE OF EACH STRIP UNDER BLOCK 'D' WAS DISCARDED

After the skins had been cut and marked each lot was divided into four groups by means of a random distribution table. Each group contained three blocks from different strips for each position (A, B, C, D) making a total of twelve blocks per treatment. The random distribution of samples minimized the between skin variation leaving two variables to be accounted for in the analysis of variance of the subsequent data, namely treatment and position.

Variables for Lot I were fatliquored stock, stock which was air dried containing no fatliquor, stock dehydrated in alcohol and stock dehydrated in acetone. Variables for Lot II were fatliquored stock, stock dehydrated in acetone, stock air dried for 48 hours, rewetted and fatliquored, and stock which was air dried containing no fatliquor.

The fatliquoring was carried out by milling the samples in twice their weight of water for fifteen minutes, neutralizing with 1 per cent sodium bicarbonate on the wet weight basis of the chrome stock for 30 minutes at 120°F. (2 to 1 float) followed by a milling in the same volume of water for fifteen minutes at 120°F. The samples were then fatliquored in twice their weight of liquor to obtain a 5 to 6 per cent level of oil at 120°F. Lot I was fatliquored with a

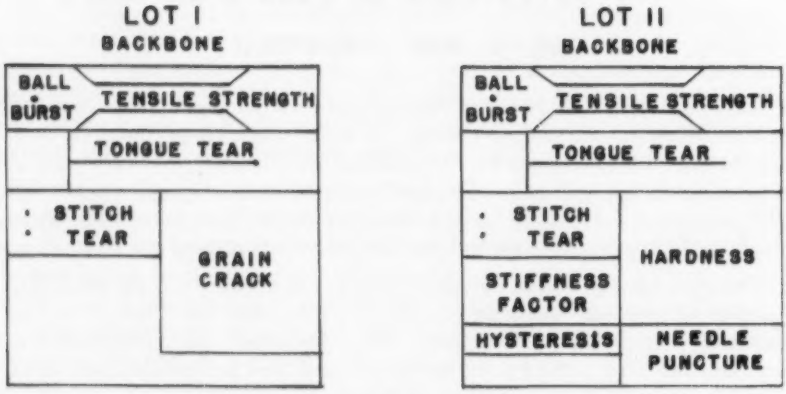
commercial sulfated neatsfoot oil fatliquor and Lot II was fatliquored with a 80 per cent sulfated cod oil and 20 per cent raw cod oil mixture. The use of the different oils will not influence the results because the experiments were set up to compare treatments within a lot and not between lots.

The solvent treated samples were treated in acetone or alcohol for 48 hours. The solvents were changed at the end of 24 hours. The air dried samples represented the chrome stock as received which was dried at room temperature for 48 hours.

After the prescribed treatments the specimens were cut for testing as indicated in the diagram in Figure II. The specimens were conditioned for at least 48 hours at 73.4° ± 1.8°F. and 50 ± 4 per cent relative humidity before testing. On the left hand side of Figure II is the plan used in obtaining ball burst, tensile strength, tongue tear, grain crack, and stitch tear specimens for Lot I. All specimens were cut parallel to the backbone. These tests were selected as a basis for our study because of their use by the leather industry for specification purpose as well as in leather research.

FIGURE II

SAMPLING OF BLOCKS FOR PHYSICAL TEST SPECIMENS



ON BLOCKS FROM LOT II COMPRESSION WAS MEASURED ON EACH BLOCK BEFORE SAMPLING

From observations and the results of testing Lot I it became apparent that what oil does in fatliquoring is related to the stress-strain characteristics of the leather more than it is to such tests as tensile strength, grain crack and

ball burst strength. For this reason Lot II as shown on the right side of Figure II included a group of tests related to stress-strain properties as well as the tests for tensile strength and others. Grain crack strength and its extension was not included in this experiment. The stress-strain tests were stiffness, compression, hysteresis, and hardness. A needle puncture test was developed and used because it was found to be more sensitive to differences than the ball burst test. All specimens were cut parallel to the backbone.

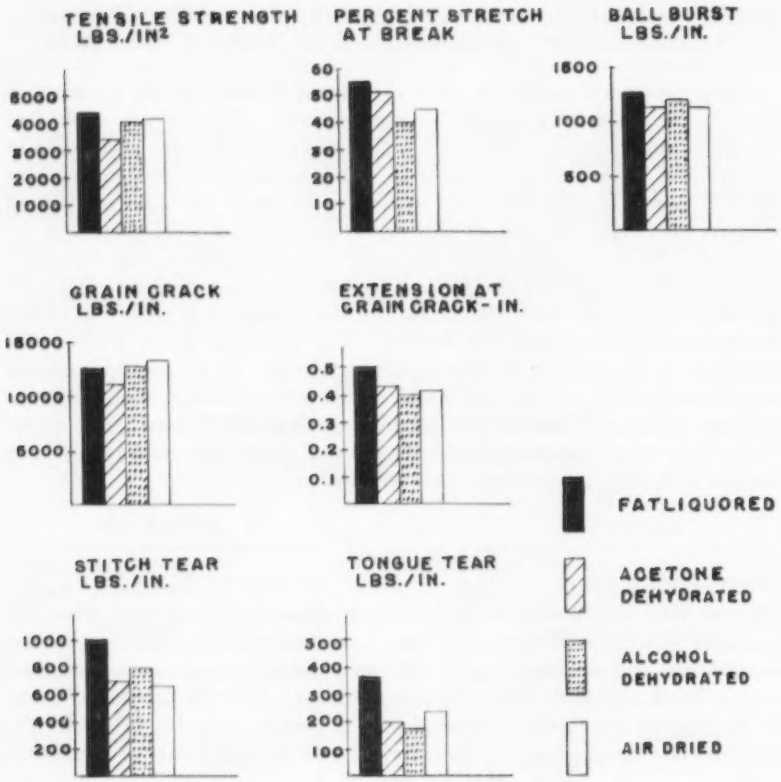
Compression was measured on a cylindrical leather sample 2 inches in diameter by means of a Schiefer Compressometer. Pressure was applied to the cylindrical specimen of leather at different intervals until the inside of the cylinder walls were just touching. The pressure was then released at different intervals. The difference in pressure obtained when plotted formed a hysteresis curve; the area of this curve, representing work done and re-recorded. Needle puncture was measured on the Schiefer Compressometer with a needle screwed into the pressure foot of the compressometer. A 1 by 2 specimen was placed under the needle and the pressure was applied until the needle penetrated through the leather. The hardness of the leather was measured on the Trionic Rubber Hardness Gauge. In measuring hardness the specimen, 2 by 2 inch, was doubled on itself to insure a more accurate reading. Hysteresis curves were obtained on a Scott inclined plane tester over a time range. The total load applied was 50 pounds, the jaw distance was one inch, and the specimen size was 2 inches by 0.5 inch.

Results

Figure III contains the average strength values of the raw data from the specimens of Lot I. The physical test measurements obtained were tensile strength, per cent stretch at break, ball burst strength, grain crack, extension at grain crack, stitch tear and tongue tear strength. The black bars represent the fatliqoured stock, the stripped bars the acetone dehydrated stock, the speckled bars the alcohol dehydrated stock and the white bars the air dried stock.

Examination of the graphs shows that the treatments have produced differences in strength and that in all cases, except for grain crack, fatliquoring produces leather with the highest strength. No trend is indicated by the other treatments. They show variation from test to test. Statistical analysis indicates whether average differences are large enough to be significantly different and whether interactions or non-homogeneous variances within the data are influencing the average values obtained. It will be recalled that two vairables, treatment and position were unaccounted for thus requiring a two factor analysis of variance.

FIGURE III
THE AVERAGE STRENGTH MEASUREMENTS OF FATLIQUORED LEATHER COMPARED TO LEATHER WHICH WAS NOT FATLIQUORED

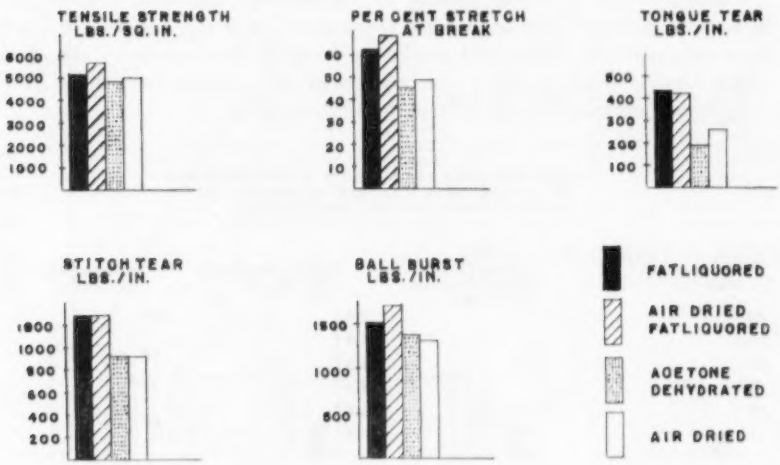


The statistics indicated significant differences between position averages for all tests except per cent stretch at break. The within data variances were analyzed for each test and the variances were found to be homogeneous in all cases except for extension at grain crack. The stitch tear and the tensile strength data showed significant treatment-position interactions. Since the extension at grain crack had a nonhomogeneous variance and stitch tear and tensile strength a significant treatment-position interaction any differences in their averages as shown in Figure III are doubtful.

The analysis of variance indicated no difference in averages existed for grain crack strength and for ball burst strength but per cent stretch at break and tongue tear strength showed significant differences. Thus the statistics have shown that the results of two tests out of seven have satisfactorily distinguished differences between treatment averages.

Figure IV contains the average strength values of the raw data obtained from the specimens of Lot II for tensile strength, per cent stretch at break, tongue tear strength, stitch tear strength and ball burst strength. The black bars represent the fatliquored stock, the stripped bars represent the stock which was air dried, rewetted and fatliquored, the speckled bars the acetone dehydrated stock and the white bars the air dried stock.

FIGURE IV
THE AVERAGE STRENGTH MEASUREMENTS OF FATLIQUORED LEATHER COMPARED TO LEATHER WHICH WAS NOT FATLIQUORED



The average strength values indicate that the treatments have produced differences. Tensile strength, tongue tear strength, stitch tear strength, and ball burst strength are higher for the fatliquored treatments than for the other two treatments. Compared to fatliquored stock apparently drying the stock first and then rewetting and fatliquoring did not materially change the stretch characteristics based on average values.

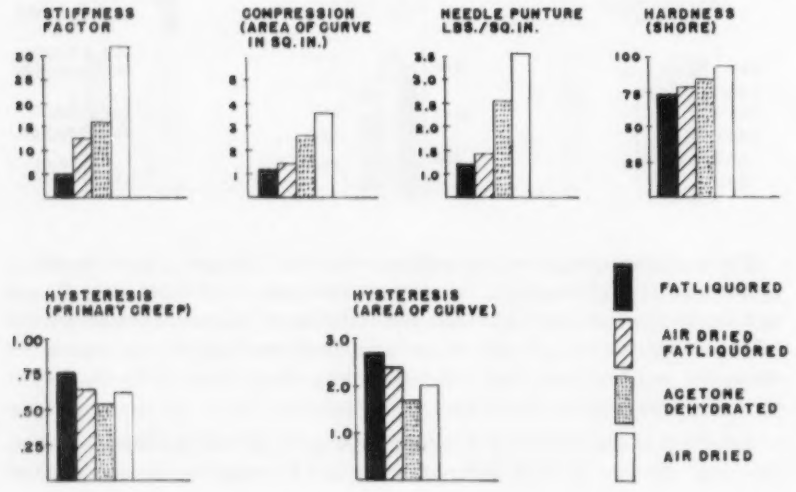
As stated in the preceding discussion of Figure III the statistical analysis indicates whether average differences are large enough to be significantly

different and whether interactions or non-homogeneous variances within the data are influencing the average values obtained. The analysis of variance indicated that no significant treatment-position interactions were present to influence the results obtained, and that significant differences between positions existed for tensile strength, ball burst strength, and tongue tear strength. There was non-homogeneous variances within the test data for tensile strength and ball burst strength which means that any significant differences between averages for treatments and positions are doubtful for these two tests.

The analysis of variance has indicated that stitch tear strength and per cent stretch at break picked out significant average differences between fatliquored treatments and the air dried or the solvent dehydrated stocks. The tongue tear test has distinguished average differences between fatliquored, air dried, and acetone treated samples.

Figure V contains the average values of the raw data obtained from the specimens of Lot II for compression, needle puncture, hardness, stiffness, and hysteresis characteristics of primary creep and area of the curve. The black bars represent the fatliquored stock, the stripped bars represent the stock which was air dried, rewetted and fatliquored, the speckled bars the acetone dehydrated stock and the white bars the air dried stock.

FIGURE V
THE AVERAGE STRENGTH MEASUREMENTS OF FATLIQUORED LEATHER COMPARED TO LEATHER WHICH WAS NOT FATLIQUORED

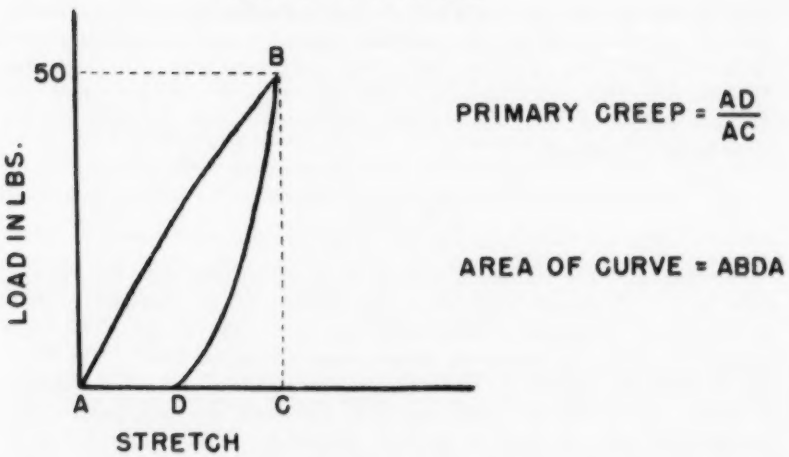


The average values indicate that treatments have produced differences. Low values are indicated for the fatliquored stock for compression, needle puncture, hardness and stiffness. These tests show a direct relationship between the tests and the four treatments averages. A relationship between test and treatment averages also exists between hysteresis characteristics of primary creep and area of the curve.

The type of hysteresis curve obtained is shown in Figure VI where the primary creep is the specimen slack and the area of the curve is the work done in deforming the specimen. The specimen was loaded to 50 pounds and then allowed to relax as the load was released.

FIGURE VI

TYPE OF HYSTERESIS CURVE OBTAINED



The statistical analysis of the data indicated that no significant treatment-position interactions were present to influence the results obtained and that significant differences between position averages existed. Non-homogeneous variances occurred within the test data for stiffness and compression and therefore any differences between the averages for treatments and positions are doubtful for these tests.

The analysis of varia ce indicated that needle puncture picked out average differences between fatliquored treatments and non-fatliquored treatments. The hysteresis characteristics of primary creep and area of the curve indicate

that they are more sensitive to average differences than the other tests used showing significant differences between three treatment means. The hardness test is the most sensitive test of the group and distinguished differences between all four treatment means.

On the basis of the statistical analysis the results show that at a force below the breaking load differences between treatment averages become more apparent. Primary creep appears to be a measure of fiber slippage or reduction of forces between fibers as indicated by the large primary creep value for the fatliquored stock compared to the other treatments. More work was required to stretch the fatliquored stock at a 50 pound load although there was no significant difference in total force (tensile strength) to break the specimens of the four treatments.

The data indicate a direct relationship in treatment averages between compression, needle puncture, hardness and stiffness. On the basis of this observation and the results of the statistical analysis, it appears that a simple test like hardness is indicative of compression and stiffness properties of leather with the advantages of being more sensitive to differences and less influenced by leather variables. The data also show treatment average relationships between hysteresis characteristics of primary creep, area of the curve, and per cent stretch at break but no relationship with these averages and the total breaking strength.

Summary

The results of the statistical analysis has pointed out that the physical tests used to determine what oils do to leather can be classified as those that are most satisfactory, satisfactory and least satisfactory. The most satisfactory tests are hysteresis characteristics and hardness. The satisfactory tests are tongue tear, stitch tear, needle puncture and per cent stretch at break. Tests such as tensile strength, ball burst strength, grain crack, compression, and stiffness were the least satisfactory in the study conducted. For the number of specimens used for each treatment the statistical analysis shows that the least satisfactory tests are not sensitive enough and that the results were being influenced by treatment-position interactions, or unaccounted within data variances.

Acknowledgment

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Discussion

Mr. Malcolm H. Battles (A. C. Lawrence Leather Co.) I want to thank Mr. Roddy for a very worthwhile paper. The significance of this paper will not be fully appreciated until further work is done evaluating some of these test methods on leather fatliquored in various ways. Mr. Roddy has demonstrated the value of these methods as a tool to studying the effect of fatliquors on physical properties. Undoubtedly, future work will use these physical measurements to study fatliquor behavor.

I will now turn the discussion over to Mr. Cares who will ask Mr. Roddy a few questions and then open the discussion to the floor.

Mr. Charles T. Cares (John Flynn & Sons, Inc.): I have only a couple of questions. I wonder if Mr. Roddy could please explain a little more fully the Schiefer Compressometer and the hardness tester, also explain a little more about the difference between air dried fatliquored and wet fatliquored stock, and whether these same tests might be applied on sheepskin.

Mr. Roddy: The hardness tester or gauge might be used in some tanneries from the standpoint of measuring the firmness or lack of firmness of the rubber rolls in various parts of the tannery. However, it is a very simple little gadget and I think that the best way to explain it would be to include a picture of the gauge in the formal paper.

In the case of the Schiefer Compressometer, it again is a physical test apparatus which enables you to measure pressures at low loads and is extremely sensitive at low loads.

You bring up a total force of about 5 lbs. on this Compressometer. If anyone would be interested in it they would be able to go back to the publications from our laboratories from the standpoint of the mechanism of bating presented by Dr. Moore a few years back. I think there is a picture of the Compressometer in that paper.

Cares: You did not find too much difference between the skins that were air dried fatliquored and those wet fatliquored?

Roddy: There is a difference from the standpoint of the hysteresis characteristics. From the standpoint of the other physical tests that we normally conduct, perhaps more samples would show a significant difference, but even

then you might find the air-dried fat-liquored stock on the average no better than the wet fatliquored stock.

CARES: Would these tests be applicable to sheepskins?

RODDY: These tests would not necessarily be applicable to sheepskins. This was done entirely on kipskin which has been shaved and is entirely different from the sheepskin.

The test could be conducted on sheepskin but perhaps an entirely different set of answers might be obtained.

ABSTRACTS

Location and it's effect Upon Structural, Chemical and Physical Properties of V. T. Leather. S. K. Sarkar, *J. Lea. Tech. Assoc. (India)* **3,** No. 1, 9 (1955). A microscopical study, chemical analysis and physical tests were carried out on leather from buffalo hide selected from the butt, neck and belly, as specified by the I.S.L.T.C. The data are given in detail with three photomicrographs showing butt, neck and belly. Fiber structure in the neck is found to be intermediate between butt and belly. Samples from the butt always give the lowest ash values and water soluble matter, and highest values for hide substance. There is a wide variation in degree of tannage from region to region in the same hide. In general fiber structure as well as chemical and physical properties vary from region to region over the surface of the tanned hide.

Entomology of Hide and Skin. B. C. Basu, *J. Lea. Tech. Assoc. (India)* **3,** No. 1, 1 (1955). Damage caused by various classes of insects is described. Those covered are; (1) Diptera, (a) Myiasis producing flies of genera Chrysomyia and Lucillia. (b) Hypoderma Lineatum (ox-warble fly) (c) Hypoderma Crossi (goat warble). (2) Anoplura (lice), coleoptera (beetles). (3) Acarina (ticks and mites). Drawings of some are included.

Soymida Febrifuga Bark as a Suitable Substitute for Imported Wattle Bark. P. S. Chowdary, K. A. Joseph and K. Padmanabhan, *The Tanner,* **10,** No. 4, 15 (1955). The bark of Soymida Febrifuga is shown to contain an average of 17.41 per cent tannins and 12.94 per cent nontannins with a pH of 4.8. Extraction is not difficult. The tannin is of the catechol type and makes good leather. The tree, also known as Indian Redwood, and in English as Coromandal Redwood, Bastard Red Cedar, Febrifuga Mahogany, Indian Mahogany, Rohan tree and Swami, tree is found in abundance in most of the forests of India.

Wattle Bark Substitutes. B. M. Das and J. B. Rao. *J. Lea. Tech. Assoc. (India),* **3,** No. 1, 4 (1955). The following tannins and blends were found to be satisfactory substitutes for Wattle. (1) Whitevalem bark, Latin name Acacia Leucophloea. Popular names, Whitevalem, Velveylam, Kikar, Tella Tuma. (2) Blend of Whitevalem and Avaram bark in proportion of 3:1. (3) Hopea Parviflora—Ironwood of Malabar. Popular names Konga, Tirpa, Kambagam and Iruppu. (4) Cassia Marginata, Popular names, Urimidi and Vakai. (5) Blend of Goran with Dhawa in ratio of 1:1. (6) Blend of Karada and Dhawa in ratio of 1:1. (7) Blend of Babul bark and Karda in ratio of 2:1. (8) Blend of Konnam and Dhawa in ratio of 3:1.

Observations on Box Calf Manufacturing. Operations between drying and finishing. F. Kahne, *The Tanner*, **10,** No. 3, 15 (1955). Operations between drying and finishing must be carried out with the same care as all other operations. After drying the leather should be stored in a cooled room, arranged for gradual absorption of moisture from air with controlled humidity. The absorptivity depends upon the previous processing of the goods, including setting out and drying. If extensively dried out in setting out or by heavy squeezing, leathers quickly become suitable for further processing. Otherwise prolonged storage is necessary. If careful control is not exercised at this stage, the unfavorable consequences must be reckoned with later. Humidity in storage rooms should be maintained at 80-90 per cent. The sawdusting, which follows, should be so carried out, that the leathers are not too wet. If too dry, it is no problem to sawdust them again. If too wet, they must be hung up to dry. This always results in uneven dryness between edges, flanks and butts, resulting in difficulty in opening up the fiber evenly by staking. If dipping is done, instead of sawdusting, the results are satisfactory if properly done. If drawn through a tank of water, the speed must be adjustable, since leathers of different size and thickness and different processing require different times. As a rough guide, dipping from 3-10 minutes may be suggested. The temperature of the water may be from 40-60°C. After dipping, the leathers are laid flat, in lots of about 100, over a wide horse covered well and left for 1-2 days in a warm room. Piling must be done with care to avoid creases, particularly in those at the bottom of the pile. Dipping in bundles is not recommended. If done, special care must be used in piling to overcome excessive creasing. Proper staking is possible only if all parts of the leather are evenly moistened. While some goods have to be staked only once, it is better to so carry it out that a slight after staking can be withstood. In this case the leathers are left lying, well covered, overnight. The flanks should be lightly staked or not at all. Properly opened leather will show no hard or firm zones when passed through the hands. The goods are next stretched by clamping or tacking. They should not be stretched excessively, especially if light willow calf. The drying automatically results in stretching. Hence flanks should not be stretched at all and heads and necks only slightly. For proper drying of stretched chrome leather, commence at about 25°C., raise the temperature within 4-8 hrs., to 35-40°C. After drying, drop again to 25°C. Before finishing, the leather should be stored a few days in a moderately warm room.

Weight Giving Property of Some Vegetable Tannins. D. Ghosh, S. K. Barat and B. M. Das. *Bull. Cent. Lea. Res. Inst.* **1,** No. 8, 6 (1955). The weight giving capacity of 5 commercial extracts was compared. The analytical figures of these extracts are given in Table 1.

TABLE 1.

	Total solids.	Total solubles.	Non-tans.	Tan-nins.
Mimosa extract	88.9%	87.6%	27.1%	60.5%
Sumach extract	89.1%	88.3%	32 %	55.3%
Quebracho extract	92.8%	88.5%	23.4%	65.1%
Myrab extract	88.8%	83.2%	33.1%	50.1%
Chestnut extract	90.8%	87.1%	25.7%	61.4%

It was thought logical to conduct the experiment on an equitan content basis, rather than on equal barkometer strengths. Hence tanning was carried out in this manner under parallel conditions until penetration was complete and all leathers had attained their maximum shrinkage values. Analysis of the leathers is given in Table 2.

TABLE 2.

Analysis of the leathers.

	Leathers tanned with.				
	Myrab.	Mimosa.	Sumach.	Quebracho.	Chestnut.
Moisture	12.5%	14.8%	12.3%	14.8%	14.8%
Hide Subs.	47%	49.4%	49%	49.2%	50%
Fat	0.12%	0.12%	0.09%	0.06%	0.12%
Water Solubles	9.4%	7.2%	9.8%	9.5%	9.6%
Insol. ash	0.42%	0.09%	0.33%	0.2%	0.35%
Degree of Tannage	65	57.4	56	53.3	50

The weights of dry leather are given in Table 3.

TABLE 3.

Per cent weight of dry leather.

	On limed weight basis.		On delimed weight basis.	
Leathers tanned with	With moisture at room temp.	Moisture free.	With moisture at room temp.	Moisture free.
Sumach	74.9%	65.2%	84.6%	73.7%
Chestnut	74%	64.4%	87.4%	76.04%
Myrab	73.6%	64.02%	87%	75.7%
Mimosa	73.1%	63.6%	82.6%	71.9%
Quebracho	69.1%	60.12%	79.4%	69.1%

The Thermal Expansion of Leathers. By R. G. Mitton. *J. Soc. Leather Trades' Chemists*, **39**, 276 (1955). Measurements of the linear expansion of chrome, vegetable-chrome retan, and vegetable-tanned leather strips were made (a) with the strips thoroughly dried and (b) when each had a constant moisture content. The damp leathers were obtained by introducing to the dry leathers water equivalent to 30, 30, and 25 per cent, respectively, of the chrome, vegetable-chrome retan, and vegetable-tanned specimens. The measurements were made by using a cathetometer to follow the elongation of the suspended leathers over a temperature range from -79.5°C. to 20°C. The coefficients of linear expansion of the dry samples were found to be 27, 30, and 29 x 10^{-6} per degree centigrade for chrome, vegetable-chrome retan, and vegetable-tanned leathers; the coefficient for the damp vegetable leather is about 50 per cent higher, but those of chrome and vegetable-chrome retan leathers are little affected by moisture content. The coefficient of cubical expansion of dry vegetable-tanned leather as determined in a dilatometer with butyl phthalate as the confining liquid was found to be 133 x 10^{-6} per degree centigrade. Coefficients of expansion transverse to the fiber axis were calculated to be 55 x 10^{-6} per degree centigrade as compared with a coefficient along the axis of 22 x 10^{-6} per degree centigrade. The direct result of heating leather articles, such as boots, is an expansion so small as to be of no importance; heating may, however, cause changes of moisture content, and these will, in turn, produce considerable changes of dimensions. J.M.C.

Vegetable Tanning. XI. Two New Methods for Determining the Acidity of Vegetable Tanned Leather. By D. Burton and L. Ma. *J. Soc. Leather Trades' Chemists*, **39,** 286 (1955). The properties of vegetable-tanned leather are a function of the amounts and kinds of the combined and free acids present. Two methods are suggested for determining the free acidity, namely, by extraction of dry leather with an anhydrous organic solvent such as methyl alcohol, and by treatment of the leather with a mixture of glycol and iso-propyl alcohol containing dissolved sodium acetate. Complete removal of free acid from 10-g. leather samples required 30 hours' extraction with methyl alcohol on a Soxhlet apparatus. The methyl alcohol extract was evaporated to near dryness and distilled water added. For estimate of free mineral acid the solution is titrated with alkali to pH 4.0, and for weak acids, to pH 7.0. The free mineral acidity can also be determined by immersing 10 g. of vacuum-dried leather in 200 ml. of a solution containing 5 g. anhydrous sodium acetate dissolved in 1 liter of a 1:1 mixture of ethylene glycol and iso-propyl alcohol and shaking occasionally for 48 hours. The extract is filtered and an aliquot titrated with 0.25 N hydrochloric acid in a solvent mixture of ethylene glycol and iso-propyl alcohol. Fifty ml. of the sodium acetate solution requires 14.2 ml. of the 0.25 N HCl and the free mineral acidity is calculated from the decrease in the amount required due to its reaction with the sodium acetate. J.M.C.

Note on the Filtration of Tannin Solutions. By H. A. Litten. *J. Soc. Leather Trades' Chemists*, **39,** 300 (1955). The S.L.T.C. official method for the filtration of tannin solutions employs an apparatus which has the disadvantage that the solution is liable to oxidation and evaporation, particularly where filtration is slow. An apparatus is proposed to overcome these shortcomings. J.M.C.

Principles of Drying. By F. E. Humphreys. *J. Soc. Leather Trades' Chemists*, **39,** 307 (1955). Problems of drying must be approached from fundamental principles, the most important being that a given amount of heat is needed to evaporate a given quantity of water. In the absence of radiant or convective heat, heat necessary for drying is derived from the air which then falls in temperature. The drying capacity of air is determined by its ability to give up heat. To obtain measurements of the drying capacity of air with wet and dry bulb thermometers, precautions must be taken. (The thermometers cannot be hung among wet leather for drying calculations.) Over the range of temperatures used in sole leather drying the true difference in wet and dry bulb temperature is a measure of the drying capacity of the air regardless of actual temperature. Increasing the rate of drying by increasing the drying capacity of the air or increasing its velocity results in dark-colored, cracky sole leather. The effect is more noticeable on unoiled than oiled leather. Increasing the temperature results in darker color and greater crackiness even if the rate of drying is not increased. Dark color and crackiness are due primarily to migration of water-soluble material to the grain surface. Increasing the temperature favors this migration by lowering the viscosity of the interfibrillary liquid. For efficient drying a definite air flow is desirable. It is preferable to increase the rate of drying by increasing the drying potential of the air than by increasing the velocity. J.M.C.

Study of the Affinity of Black Wattle Extract Constituents. Part III. The Bisulfite Reaction on Wattle Polyphenols. By D. G. Roux. *J. Soc. Leather Trades' Chemists*, **39,** 321 (1955). Paper chromatography and chromatography employing columns with collagen powder as adsorbent were used to study the behavior of d-catechin, black wattle and also quebracho polyphenols during the well-known bisulfiting process. Paper chromatography indicated that d-catechin is readily converted to addition compounds by heating with 5 per cent bisulfite. Some d-catechin is unchanged and some transformation to

d-epicatechin occurred. The addition compounds formed show greatly reduced and very low affinity towards cellulose. Black wattle polyphenols (tannins) behave similarly. The more astringent tannins characterized by low Rf react preferentially to the more mobile constituents. This preferential action also occurs slowly at room temperature and under commercial conditions for both wattle and quebracho extracts. The bisulfite-tannin addition compounds still show fairly high, although considerably reduced affinity for collagen. The preferential action of the bisulfite for the astringent colored fraction explains the increase in solubility and reduced astringency. The concept that bisulfite reduces the particle size of tannins should be re-examined. The lower gelatin number as well as the reduced fixation recorded by previous workers for quebracho and black wattle upon bisulfiting may be explained in terms of the reduced affinities of the astringent fractions of these extracts for collagen. J.M.C.

A Study of Mold Growth on Chrome-Tanned Leather, and the Associated Changes in Physical Properties. By R. G. Mitton and J. N. Turner. *J. Soc. Leather Trades' Chemists*, **39**, 343 (1955). The growth of mold on chrome leather was studied. Before storing samples at either 87 per cent or 100 per cent relative humidity, *p*-nitrophenol was added to some in amounts sufficient to repress mold growth without inhibiting it entirely. Some of the samples contained their natural fats, together with that added as fatliquor at the tannery. To some, cod oil or a mixture of cod and neat's-foot oil was added. To degreased samples, mineral oil was added in one instance, and a mixture of cod and neat's-foot oil in another. The water extract of some samples had a pH of 4.4, and the rest, pH 5.9. Storage was at 24°C. or 30°C. and for periods which in one experiment were 3, 6, and 9 months and in another 12 months. Mold grew freely on the samples stored at saturated atmosphere but less vigorously at the lower humidity. The extent and nature of the molds showed considerable dependence upon the pretreatment of the samples and on the storage conditions. One mold not previously reported on leather, *Penicillium islandicium Sopp.*, grew freely on samples containing mineral oil, and produced deep red stains. The physical properties of the leathers were changed little by the mildew growth and such changes as occurred were due to modifications of the oil rather than of the leather fibers. In particular, tensile strength was unaffected by mold growth. J.M.C.

The Concept of Olation. By A. Küntzel. *J. Soc. Leather Trades' Chemists*, **39**, 359 (1955). The concept of olation, originally developed by Stiasny, differentiates between basicity and olation. The term olation refers to the aging process of chromium compounds after addition of sodium hydroxide, characterized by (1) becoming acid-resistant, (2) decomposition into products of smaller particle size, and (3) a fall in pH value. This concept is restricted to soluble basic chromium salts, which are related to two different types of insoluble chromium hydroxide: the non-olated basic salts are the precursors of α-chromium hydroxide, and the olated basic salts are the precursors of β-chromium hydroxide. The effect of the kind of alkali used on the process of olation is discussed. The author's olation theory is offered to explain the decrease in particle size which occurs during olation. The paper is to a considerable extent a criticism of a paper by S. G. Shuttleworth on olation (*J.S.L.T.C.*, **39**, 123 (1955)). J.M.C.

PATENTS

Shoe Impregnites. U. S. Pat. 2,719,794. Harold P. Averill, Aberdeen, Md., Ralph W. Peakes, Washington, D. C., and Frank Carl Whitney, Edgewood Arsenal, and Herbert A. Scruton, Baltimore, Md., assignors to the United States of America, in trust, as represented

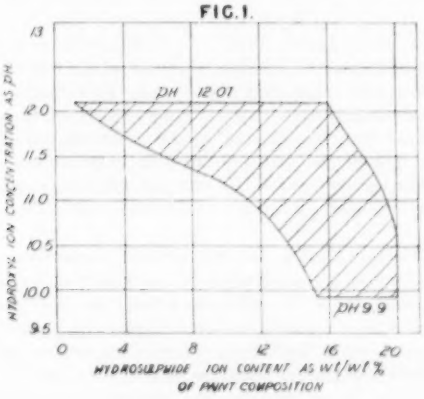
by the Secretary of War. 1. A composition of matter for treatment of leather to render it resistant to penetration by mustard gas, Lewisite and like vesicants which comprises about 40 parts by weight of paraffin, about 40 parts by weight of ester gum, and about 20 parts by weight of neat's-foot oil.

Deposition of Polymers Into Leather. U. S. Pat. 2,721,145. Nicholas D. Cheronis, Brooklyn, N. Y. 1. In a method of impregnating leather to deposit within the interfiber space therein sufficient elastomeric material to increase substantially the water resistance of said leather without, however, decreasing the water vapor permeability thereof to an objectionable degree, the steps which comprise adjusting the pH of the tanned leather to within the range of about 3 to about 5.5, preconditioning said leather by depositing therein several per cent, by weight, of at least one member selected from the group consisting of sulfated and sulfonated animal and vegetable oils, washing said leather in water, draining off the wash water, rolling the leather while wet and maintaining it rolled for at least 24 hours, then tumbling said leather while warm with a warm emulsion containing, by weight, from about 2 per cent to 5 per cent of a polymerized elastomeric material, from about 90 per cent to 95 per cent of water, from about 3 per cent to 5 per cent of a volatile water-immiscible predominately non-polar organic solvent, from 0 per cent to 1.5 per cent of a plasticizer, and from about 0.4 per cent to 0.7 per cent of an emulsifier, said emulsion containing from about 2 per cent to about 5 per cent, by weight, of solids and having a pH between about 6.5 and 8.5, the leather being tumbled until the emulsion is substantially exhausted of depositable solids, and then drying said impregnated leather.

Waterproofing Leather. Brit. Pat. 706,719. Dow Corning, Ltd. Apl. July 27, 1951. Leather is rendered water-repellent by contacting it with a methyl siloxane copolymer (*see* Group IV (a)), preferably in solution in a volatile solvent, e.g. petroleum hydrocarbons, benzene, toluene and xylene, so that the leather contains from 0.1 to 60 per cent. by weight of the leather of the copolymer. Other polymers, such as liquid dimethylsiloxanes, methylphenysiloxane resins, liquid methylphenylsiloxanes, stearylsilsesquioxane, butylmethylpolysilane, phenymethylpolysilane and stearylmethylpolysilane, may be added to the copolymers.

Depilating Skins. Brit. Pat. 706,746. H. Zaleman. Apl. April 4, 1952. A depilatory composition comprises calcium, barium, strontium, aluminium or magnesium sulphide

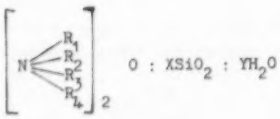
FIG. 1.



or hydrosulphide and calcium, barium or strontium hydroxide, with or without a soluble calcium salt in the case where the sulphide and hydroxide are of different metals and together with a soluble calcium salt other than calcium hydrosulphide in the case where the sulphide and hydroxide are of the same metal, in an aqueous suspension in which the hydrosulphide ion content and the hydrogen ion concentration are within the range defined by the shaded area (Fig. 1). In some instances the addition of a soluble calcium salt, such as calcium chloride, or an acid, such as hydrochloric acetic, hydrobromic, formic, lactic, nitric, nitrous, tartaric or citric acid, may be necessary to give a pH in the desired range, but it is desired to choose the ingredients of the composition and their proportions such that the desired hydroxyl ion and hydrosulphide ion concentration is produced without the need for depressing the pH. The desired consistency may be adjusted by the addition of an inert filler such as kaolin.

Tanning Agents. Brit. Pat. 709,581. O. C. Jekel. Apl. Dec. 10, 1951. Natural tannins are reacted with boric acid in the presence of an organic solvent to form compounds useful as tanning agents. Suitable solvents are methyl, ethyl, *n*- and isopropyl, butyl, amyl, allyl and propargyl alcohols, formic, acetic, and propionic acids, esters of these alcohols and acids, acetaldehyde, acetone, methyl ethyl ketone, ethyl ether, and mixtures thereof. A preferred solvent is a mixture of ethyl alcohol, acetic acid, ethyl acetate, and water in equilibrium proportions. Reaction takes place at normal temperature or with warming, and the compounds formed may either be used in solution in the organic solvent or recovered by evaporation and recrystallisation from ethyl acetate. In examples boric acid is added to organic solvent extracts of oak bark and pomergranate tree bark. Higher organic acids, alcohols, and esters, e.g. lauric acid, myricyl alcohols, and beeswax, may be added to the solution; other additives mentioned are softening, water-proofing, coloring, bactercidal, insecticidal and insect-repellent substances. Before use the solution may be acidified by adding up to 15 per cent. of formic, acetic, or propionic acid.

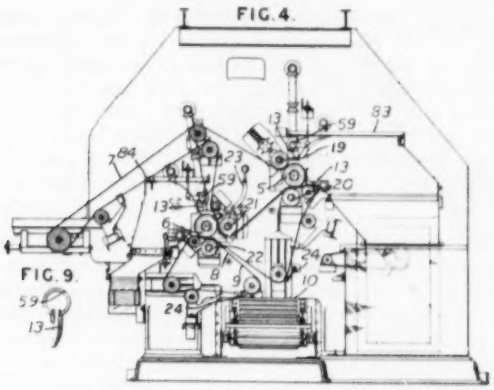
Leather. Brit. Pat. 709,634. Crosfield & Sons, Ltd., J. Apl. Feb. 20, 1951. Organic nitrogen base silicates of the general formula



wherein R_1 and R_2 are independent substituent groups selected from hydrogen and alkyl, hydroalkyl, aminoalkyl, amidino, cycloalkyl, aralkyl and aryl groups, R_3 being an independent substituent selected from hydrogen and the aforesaid groups and R_4 being an independent substituent group selected from the aforesaid groups other than alkyl or R_3 and R_4 being both $-CH_2-$ groups forming part of a heterocyclic ring structure which includes the nitrogen atom, X is a number varying from 0.5 to 4 and Y is a number varying from 0 to 15, are used for dehairing leather.

Treating Skins. Brit Pat. 711,864. Tanners Research Corporation. Apl. July 26, 1951. Natural matter is detached or severed from skins by subjecting one or both sides of the skins to one or more fluid jets under sufficient pressure to effect such detachment. The skins are fed to the apparatus on perforated conveyer 7, pass round cylinder 5 where the skins are unhaired by means of jets of liquid (e.g. water) from nozzles 13, then on conveyer 8 to cylinder 6 where flushing is effected by means of similar jets, and finally out of the apparatus on conveyers 9, 10. The nozzles associated with each cylinder are arranged in two

rows converging on to the skin; the jets operate intermittently, the first row on the leading



portion of a skin and the second row on the trailing portion. Grip rolls 19, 20, 21, 22 hold the skins taut in contact with the cylinders. Liquid sprays 23 or scrapers 24 serve to detach the skins from the conveyers. Solenoid-actuated valves controlling the rows of jets are opened and closed in sequence by an electronic control device actuated by a photo-electric cell whose beam is interrupted by passage of a skin (*see* Group XL (b)). The diameter of the jet orifice is 1/16-1/2 inch and liquid pressures of 150-1,200 lbs. per sq. in. are used. Nozzles as shown in Fig. 9 may be arranged in echelon formation in order to maintain a constant pressure over the area of impact of the liquid with the skin. The angle of contact between the jet and the skin may be 45 degrees to 75 degrees when the jets are in the same direction as the movement of the skin, and 25 degrees to 60 degrees when the directions are opposed. Liquid is supplied to the jets from headers 59 which are adjustable in order to vary the distance of the nozzles from the skins and the angle of contact of the jets. Used liquid and waste material removed from the skins are collected in hoppers 83, 84. Reference is made to the use of gaseous fluids, e.g. air.

Deliming Hides and Skins. Brit Pat. 714,165. Benckiser, J. A. Apl. July 23, 1951. Hides or skins are delimed by treatment in a bath of or containing unrefined citric acid fermentation liquor produced by fermenting carbohydrates. The bath may also contain citric acid fermentation liquor from which calcium citrate has been precipitated or liquors from which tartaric acid or citric acid or salts thereof have been crystallized out; lactic acid, acetic acid, tartaric acid, ammonium sulphate, pyrophosphates, polyphosphates, or metaphosphates may also be present. The fermentation liquor may be used as an additional deliming component in artificial leather heating. Examples describe the treatment of a goat skin and a calf skin.

Treating Animal Pelts. Brit. Pat. 714,829. Wool Industries Research Association. Apl. Jan. 14, 1953. Animal pelts are treated with gaseous chlorine or bromine in vacuo, the material being cooled after drying prior to insertion in the vacuum chamber. The pelts may be conditioned to a moisture regain between 4 and 8 per cent. and cooled to a temperature between 35° and 45°F. before the gaseous treatment, and afterwards neutralized with sodium sulphite. Specifications 417,719, 475,742 and 493,098, [*all in Group* IV], are referred to.

Leather-tanning Materials. Brit. Pat. 716,664. Wurm, G., (trading as Wurm Leder fabrik G. (Firm of).). Apl. May 26, 1952. Tanning materials are produced by adding to the waste liquors from the sulphite digestion of the wood of deciduous trees, especially beech, hot concentrated viscose spinning bath wastes containing alkali metal suphites, separating calcium sulphate, reacting the solution with cation exchangers in the hydrogen form, concentrating and heating to 150°C. the solution now containing free ligneous acids, particularly sulphonic acids, adding stabilizers, e.g. 2 per cent. naphthalene sulphonic acids or their homologues, and finally atomizing the solution, when the solids content is 40-60 per cent. by weight, e.g. by rotating a disc, in a drying tower to produce a voluminous powder. The hygroscopicity of the powder may be reduced by adding 0.2 per cent. Turkey red oil. The tanning materials may be dissolved in water and dialyzed through a viscose membrane.

Artificial Leather. Brit. Pat. 730,221. Eberle H., and Saladin, O. Apl. Nov. 18, 1952. Artificial leather, which is permeable to air and steam, but sufficiently impermeable to water, is produced by covering a fibrous support with a layer of a dispersion of an olefinic polymer containing 5—30 per cent water and evaporating the volatile components from the layer by heating to a temperature higher than 120°C. to produce a network of pores in the layer. The resin layer may be gelatinized. The fibrous support may be highly absorbent woven or knitted fabrics, fleeces, or paper and the olefinic polymer may be polyvinyl chloride, copolymers of vinyl chloride and vinyl acetate, or mixtures of vinyl and methacrylic acid ester polymers. The coated support may be subjected to electric spark discharge to increase the porosity, and may be given a final coating of non-porous olefinic polymer plastisol, which is also rendered porous by electric spark discharge treatment. Inorganic or organic gas-releasing compounds may be dissolved or dispersed in the aqueous dispersions, or their solutions may be used in place of water for making the dispersions, to release gas by heat or added acid during the entire drying and gelatinization treatments, with simultaneous evaporation of water, to increase the porosity. Suitable compounds are sodium bicarbonate, or ammonium carbonate, bicarbonate or carbamate. Fibrous material of animal, vegetable or synthetic origin, e.g. wool dust, cotton, cellulose, shredded or powdered leather, staple length being 1 mm., may also be added to the dispersions to increase the porosity. The porous product may be impregnated to increase the water repellency by oiling or greasing with cream or with aqueous paraffin dispersions. In examples: (1) extruded polyvinyl chloride is mixed with pigment, dioctyl phthalate and a mixture of higher molecular weight hydro-carbons and the mixture homogenized in a three-roll mill to form a pasty mass *a* which is stirred with water until a spreadable mass *b* is obtained; the mass *b* is coated on cotton dried at 140°C., gelatinized at 150—175°C., grained, subjected to electric discharge, and greased; the resulting product is dried at 140°C., over-coated with the pasty mass *a*, gelatinized at 175°C., grained, and again subjected to electric discharge; (2) a mixture of polyvinyl chloride and polymethyl methacrylate is used; (3) a mixture of a vinyl chloride-vinyl acetate copolymer and polyvinyl chloride is used; (4) the pasty mass *a* of (1) is stirred with an aqueous solution of ammonium bicarbonate to form a spreadable dispersion. (5) a pasty mass similar to *a* of (1) has cotton shearing dust added, and is stirred with an aqueous solution as in (4).

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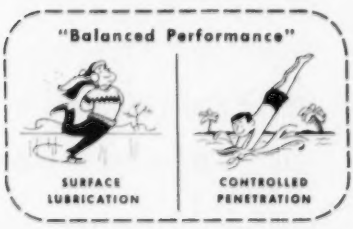

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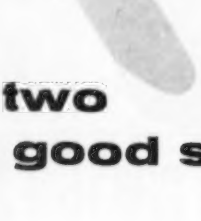

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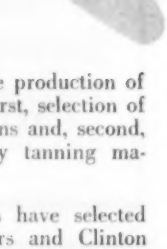

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